

# metal finishing

PAINT APPLICATION, ELECTRODEPOSITION, VITREOUS ENAMELLING,  
GALVANIZING, METAL SPRAYING and all METAL FINISHING PROCESSES.

Founded 1904 as THE  
INDUSTRIAL FINISHING JOURNAL

Vol. 7 No. 76 (New series)

APRIL, 1961

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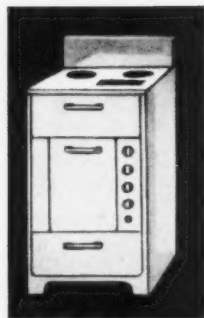
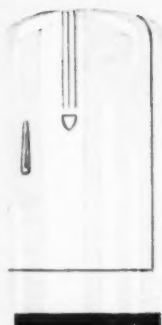
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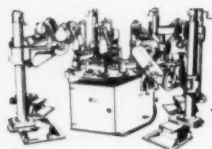
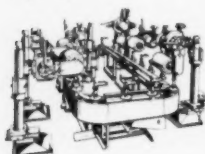
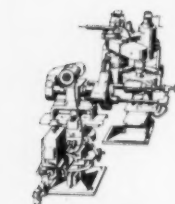
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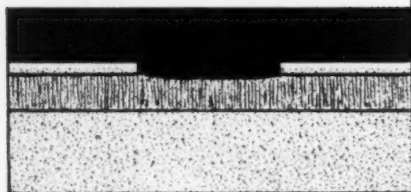
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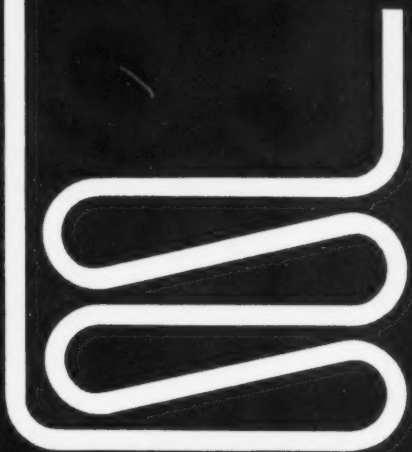
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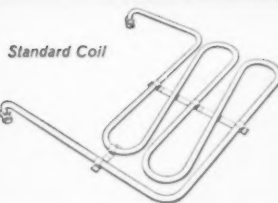
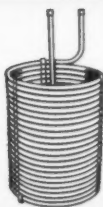
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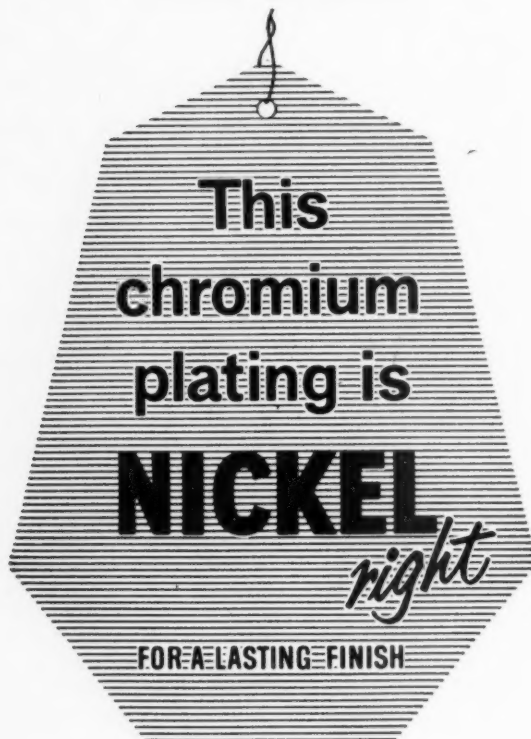
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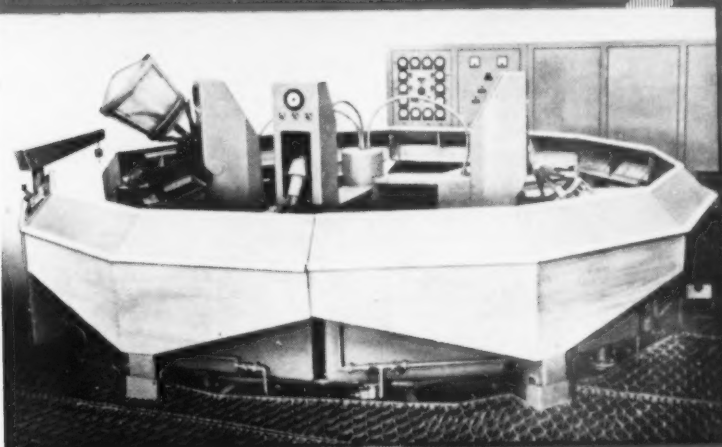
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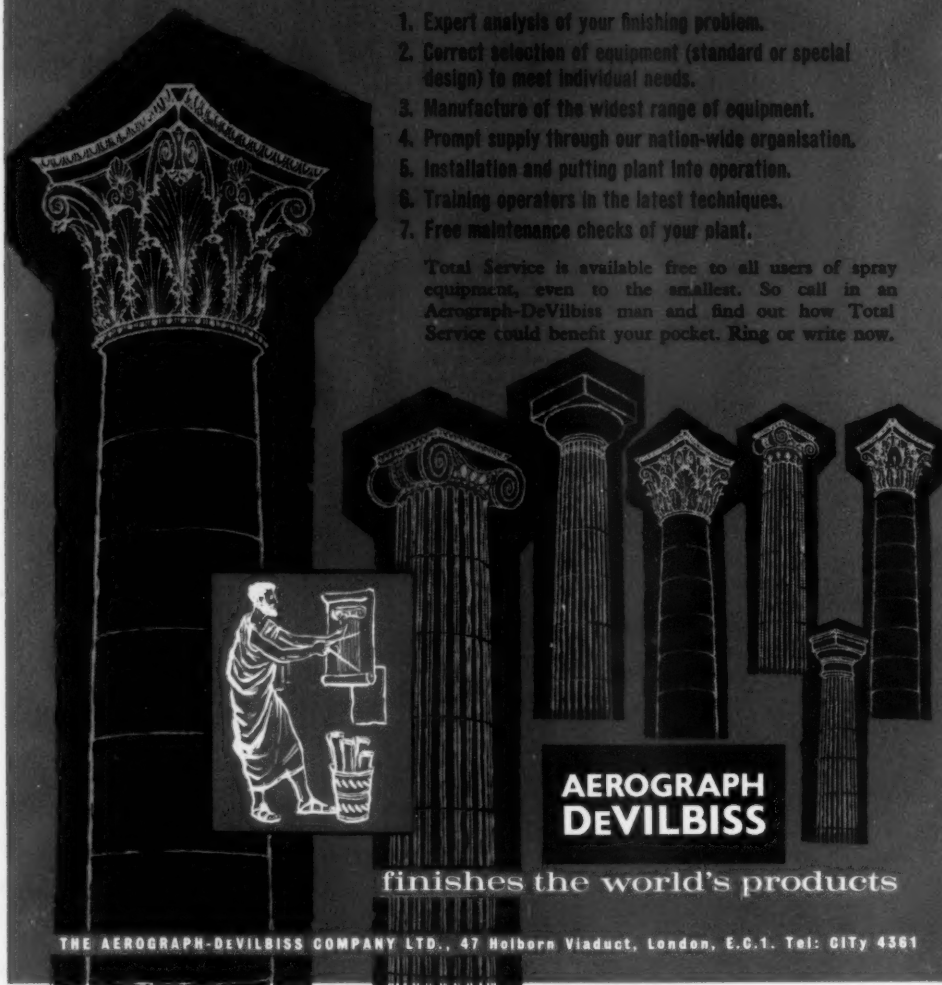
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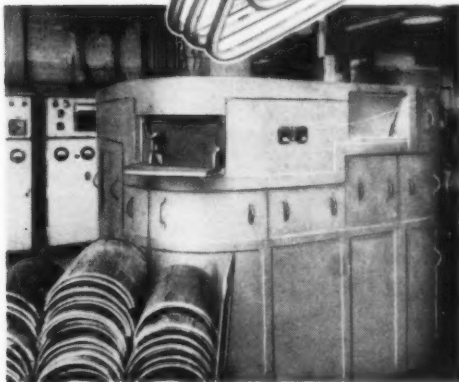
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# metal finishing Journal

April, 1961



Vol. 7, No. 76 (New Series)

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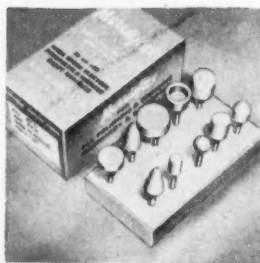
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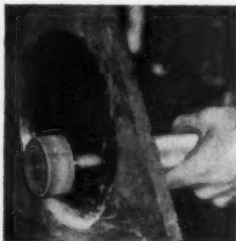
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## THE ODD ONE

THE controversy regarding the quality of the paintwork, plating etc. on motor cars shows no sign of abating and continually in the correspondence received by this Journal this controversy continues. It is perhaps unfortunate that, unjustly, the motor car has had to bear the brunt of most of the adverse comments, as our mail shows that the manufacturers of domestic equipment, such as cookers and washing machines have no room for complacency.

We know that the production of such goods has been virtually revolutionized since the war, and it is only because of this that prices have been kept at a level that compares more than favourably with those reigning 20 years ago, but the considerable capital expenditure on improved production equipment and the increasing use of automation should, in theory at least, ensure not only a high standard of quality but also that this quality is maintained.

There is no doubt that the raw materials, in the form of improved electroplating solutions, paints, etc. are in the hands of the manufacturers, but the quality of some finished goods still leaves much to be desired and brings in criticism both from home and abroad. And this is in spite of the fact that so much has been written and said regarding the necessity for this country to increase its export trade, a factor more than ever to be considered in the light of the recent "Economic Survey." One must look, therefore, for some other reason for the apparent failure of new finishing materials and plant to produce the results claimed for them.

We are forced to conclude, that it can only be the human factor that is deficient, as, in the last analysis it is human skill and the care that the operative puts into his work that determine the final quality of a product. It can only be assumed, therefore, that to some extent it is shortcomings in inspection procedures that make a major contribution to one of industry's main problems, the net result being a reduction in the appeal of a product to customers. There should be no reason for even one finished article to be delivered with the plating already showing signs of corrosion, or with defective vitreous enamel or paint, and the importance of stopping a product getting through inspection in such a condition should not be underestimated, as it is the "odd one" falling into users' hands that later becomes the "odd hundred or so" in conversation due to the natural frailty of the human to magnify his troubles.

There is a lack of pride on the part of the average operative in industry in the product he is helping to manufacture and this is a weakened link in what should be a perfect chain. Let us by all means give prominence to the cry of "export or die," but managements should encourage labour to make a pledge to take a keen interest in their job and to make a good job of that job. Although the system of bonus payments etc. in use today does not help to achieve this, it should not be impossible to find some means by which traditional British pride in craftsmanship can be revived to become a major selling point as it was not so many decades ago.

# Talking Points

by "PLATELAYER"

TOPICAL COMMENT  
FROM THE MAIN  
LINES AND SIDE  
LINES OF METAL  
FINISHING

## LAYING THE POISON

THE efforts of the Borgias were as nothing to those of industry, which unleashes into our homes a vast stream of active potential poisons for such mundane purposes as cooking the lunch, getting spots out of clothes, or polishing the knocker! Last year these products killed nearly 8,000 people, and hospitalized 70,000 more.

One of the troubles is that as often as not the materials are proprietary, and the doctor has no knowledge of the exact nature of the toxic agent involved, or the antidote. It is therefore good news that a scheme to set up a Poisons Reference Centre is being contemplated by the Ministry of Health.

The plating industry uses large quantities of very poisonous materials, and takes appropriate precautions; hence the accident rate from these causes is low. It seems ironical that the hazards which platers might meet at home from substances in innocuous looking packages which often give no indication of their danger or of the antidotes to use in the event of them being accidentally ingested, might be greater than those encountered at their places of work.

## HIT OR MISS METHOD

A RECENTLY described method of applying a corrosion-resisting coatings of titanium to other metals on the Continent consists of projecting titanium balls at an oblique angle against the surface to be treated by means of compressed air.

The procedure appears to involve the expenditure of a large amount of energy for a limited result, and in this respect it reminds us of the story of the man who came back repeatedly to a chemist's shop for large quantities of mothballs. After a time, the chemist enquired whether he was not finding them effective in destroying the moths. "Yes, they are extremely effective," came the reply, "but I can't manage to hit them every time."

I trust I may be pardoned for saying that it is unlikely that the process will make much impact on the finishing industry.

## COATINGLESS ELECTROPLATING

A RECENT news item in "Plating" announces that the Firestone Steel Products Company has a new finish for wheels, and that "tests to date on the new electroplating process show it to be superior to paint finishes and will save fleet

owners more than 50 per cent in re-finishing and maintenance costs over the life of the wheel."

This in itself is not a very remarkable piece of information—after all, new wonder processes in the plating field are discovered almost daily. What is intriguing, however, is that the statement goes on: "Since it is not a coating, this new finish cannot chip or rust and makes the wheel impervious to corrosion from road salt and other acids." In spite of having been conditioned to electroless plating, I find it startling now to be confronted with coatingless plating.

On the other hand, perhaps the information is to be taken with a grain of (road) salt. Some technical journals are like modern buildings; the fronts are often magnificent, but the backs do not bear close inspection.

## RESEARCH REPORT

OUR work, and that of others before us, with surface electron microscopic studies, shows that brightness is associated with absence of surface projections."—*From a paper in the Spring 1961 Transactions of the Institute of Metal Finishing.*

And also, no doubt, with absence of surface dullness.

## MISUNDERSTANDING

THERE are two misconceptions about stainless steel, according to manufacturers participating in an exhibition at the Mayfair Rooms, Bryanston St. One is that it is stainless."—*Daily Telegraph.*

The man-in-the-street is very tedious. He seems to misunderstand both technical terminology and plain English.

Stainless steel just isn't stainless—  
Don't let sales talk take you in ;  
Hot tin roofs are built of iron,  
Tinfoil isn't made with tin.

Silver coins are struck from Monel,  
Not a trace of real Ag ;  
Coppers are no longer copper—  
Still, it's all good "brass" to me !

Shockproof, mothproof, waterproof, bombproof—  
Don't believe a word you see ;  
Rustless, noiseless, dustless, priceless,  
Fings ain't wot they're sed to be !

# THE ACCELERATED WEATHERING OF ORGANIC FINISHES\*

## A Critical Study

By A. A. B. HARVEY†

*Accelerated weathering cycles consist of exposure to light, moisture and elevated temperature, either separately or in combination. To these agents are sometimes added refrigeration and chemical attack usually by sulphur dioxide. The spectrum of the sources of radiation is compared with that of average sunlight and the effect of the various spectral regions on an organic film discussed. It is concluded that, in natural weathering, wavelengths below 300mμ play no part. Where artificial sources include wavelengths shorter than 300mμ, selective absorption of shorter wavelengths is the exception. The study of results shows that the combination of condensation and irradiation is a more effective agent of destruction than either separately. Temperature plays a part in film degradation but any rise above 40-60° C. may produce spurious effects in accelerated cycles.*

*The accelerated weathering systems used in the United Kingdom which employ a carbon arc, a mercury arc and a twin carbon arc with subsidiary chemical treatments, are compared and the degree of correlation claimed with natural weathering assessed.*

*The valid use of accelerated weathering as an accurate predictor of film life or merely as a sorting agent among known compositions is discussed. The conclusion is reached that, as the analyses of natural climatic conditions given in the paper show a great variation from year to year, even at the same place accelerated, weathering results must be interpreted against a wide background of natural exposures. Control samples simultaneously exposed are essential. No life prognostication can be safely made. The safe limit acceleration seems to be of the order of twenty times, but higher values than this are claimed with good correlation with natural weathering.*

THE life of a modern organic finish exposed to service out of doors may, except under the very worst conditions, be measured in terms of years. It is, of course, necessary to qualify that statement with the proviso that the finish has been applied according to the dictates of good practice to a properly prepared and suitable ground. This degree of service durability makes it important to the paint manufacturer and user alike to have at his disposal a means of assessing the quality of finishes in a shorter time than needed by field exposures. The manufacturer has to decide quickly whether certain lines are safe to market or whether promising new materials will live up to their promise. The paint user has to select between competing materials. If he is concerned with the painting and maintenance of permanent structures or costly equipment, quite minor differences in durability may give rise to large economic effects. It is to satisfy the needs of both sides of the paint industry that so much attention has been given to the development of accelerated weathering cycles for paint testing.

The aim of such weathering cycles is to produce, in a few weeks or days, the effects of possibly several years exposure of a paint film to the weather

and to reproduce all the complex modes of failure of a weathered organic finish. Probably the best definition of the purposes of accelerated weathering is better known in North America than in England, since it was furnished by H. G. Arlt<sup>(1)</sup> for the American Society for Testing Materials. He defines the aim of accelerated weathering as "To reproduce in a single test all the factors encountered under service conditions and to produce failures in the organic finish resembling those found in service." As any definition should be held to exclude that which it does not specifically include, we may read into this a prohibition of the use of all factors which play no part in the deterioration of a finish in service, or the use of the natural agents of degradation at such a level of intensity that their mode of action is materially altered. In short, the same changes that occur in natural weathering must be induced in the film by the same agents, but more rapidly.

Since the simplest of applied finishes, a one-coat clear-varnish system, consists of at least two layers, the substrate and the applied film, the study of isolated paint films or incomplete finishing systems, can only yield limited and often misleading information on durability. The assessment of durability by either accelerated or natural weathering can only be valid when conducted on the multiple layer systems of commercial practice, so that the

\*A paper presented to the North West Branch of the Institute of Metal Finishing.

†Director of Technical Services, Sunbeam Anti-Corrosives Limited, Central Works, West Molesey, Surrey.

chemical and physical action of layer upon contiguous layer is taken into account.

### History of Accelerated Weathering Tests

A complete and detailed historical review of accelerated weathering would occupy more space than could be justified in a paper of this nature as two such reviews and bibliographies have already been made; one down to 1949 by the Staff of the Paint Research Station<sup>(2)</sup> and down to 1953 by A. Kutzelnigg.<sup>(3)</sup> Both these give details of the various accelerated cycles which have been proposed or are in use.

To America we owe the series of remarkable papers of H. A. Nelson and his collaborators, the first of these<sup>(4)</sup> published in 1922, is universally regarded as the pioneer paper on this subject.

In England the study of accelerated weathering commenced with the railway companies and has been taken up in later years by the motor industry and the Paint Research Station. These three agencies have each produced an accelerated weathering cycle which is in active use as a part of purchasing specifications. The classical English paper summing up twenty years experience in this field is that of Dunkley and Fancutt,<sup>(5)</sup> given to the second F.A.T.I.P.E.C. Congress in 1953.

All this work has resulted in the accumulation of a mass of information, many cycles of accelerated weathering have been proposed and many discarded, but there are now two main streams of work, one using carbon arcs as the source of irradiation and the other a mercury arc in a quartz envelope. Both these show variations by different users in the length of time for which the wet film is irradiated, the humidity of the atmosphere during dry irradiation, the use of chemical breakdown agents, principally sulphur dioxide, and the use or omission of refrigeration as part of the cycle.

### Paint Film Failure

A wet paint film applied to a substrate dries according to its type by loss of solvent, by oxidation, by polymerisation or any combination of these three. From the moment the film is applied, changes commence which continue throughout the life of the protective system.

The simplest case is that of the solution type of paint such as nitrocellulose, this dries primarily by loss of solvents. The dry film contains plasticizers whose loss by migration to the surface or evaporation causes embrittlement. Plasticizer loss is greatly accelerated by exposure to high temperature. At the same time, the material of the film may cross link under the influence of oxygen and again hardness and embrittlement result. These changes, together with a possible decomposition

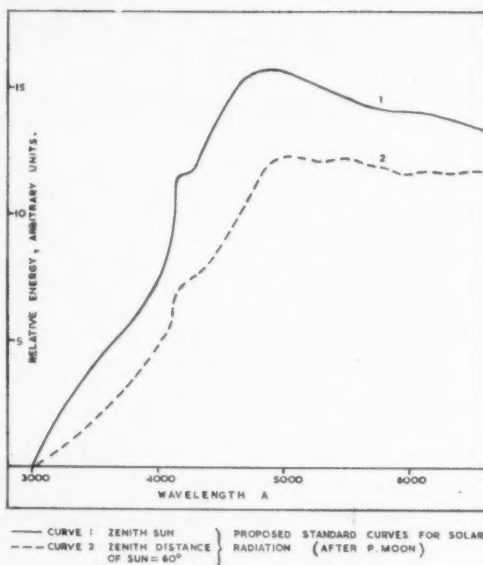


Fig. 1.—Distribution of energy in the spectrum of sunlight. (P. Moon<sup>(9)</sup>).

of the film-forming materials, are the typical changes in those paints which dry by evaporation.

The more complex films formed by drying oils, oleo resinous and similar varnishes, dry mainly by cross linking induced by the formation of peroxides of the unsaturated acids. The great variety of cross linkages so formed may act in the early life of the paint film as a lattice in which are held some of the partially oxidized varnish and unsaturated oils. Like the plasticizers of the solution-type paints, these may be capable of a limited mobility or may be extractable by water. In any case, their unsaturated nature renders them liable to further cross linking with the formation of new products and consequent embrittlement of the film. A discussion of the mode of drying of these paints was given by S. H. Bell.<sup>(6)</sup>

Since that date epoxy resin and similar varnishes drying by combination of molecular species already within the varnish have come into use for outside exposure. The degree of combination may be halted at any convenient stage of cure, leaving much less opportunity for breakdown of the type described.

Since the mechanism of film failure of the various types of finish varies so greatly, there is little *a priori* reason to suppose that a single cycle can be found to accelerate failure to the same degree or even to produce the precise type of failure found in each case after prolonged outdoor exposure.

### The Agents of Breakdown in Natural Weathering

For the purposes of designing accelerated weathering cycles, the following factors have been isolated as the main causes of breakdown of paint films exposed to natural weathering:

- (1) Absorption of radiant energy from sunlight.
- (2) Elevated temperature and thermal shock caused by rapid temperature changes.
- (3) Water in contact with the paint film.

For certain specialized types of exposure in industrial environments an addition to these may be made of—

- (4) Industrial atmospheric contaminants, particularly sulphur dioxide.

#### (1) Absorption of Radiant Energy

Sunlight falling on a paint film consists of various wavelengths of electromagnetic radiation between the near ultra-violet through the visible spectrum to the infra red. This band of frequencies is the most active in promoting chemical change as it can be absorbed by the valency electrons of the atoms constituting the paint film and activate them to the level where chemical action can proceed. The energy available in the radiation to promote chemical reaction is measured by the quantum energy per photon and this is therefore of fundamental importance. The higher the energy absorbed per photon of radiation, the greater is the possibility of chemical action arising from the activation of the molecule.

The energy  $U$  per photon of electromagnetic radiation of frequency  $\lambda$  is given by the equation:—

$$U = \frac{hc}{\lambda}$$

where  $h$  is Planck's Constant  $6.547 \times 10^{-27}$  erg seconds, and  $c$  is the velocity of light  $3 \times 10^{10}$  cm. per sec.

The energy required to rupture most of the common organic valency bonds is that carried by a wavelength of 300  $m\mu$  or less. Gusman and Stroupe.<sup>(7)</sup> This wavelength is in the near ultra-violet and marks the approximate lower limit of wavelength found in sunlight falling on the earth's surface. When considering the effect of light on paint films subjected to accelerated weathering cycles, it is necessary to examine carefully the ultraviolet region since radiation in the visible and infrared will not, in general, have sufficient energy per photon to effect materially the life of the film. The results of J. S. Long<sup>(8)</sup> that the durability of an alkyd film is proportional to the area under the transmission curve between 300 and 400  $m\mu$  wavelength are in direct confirmation of this.

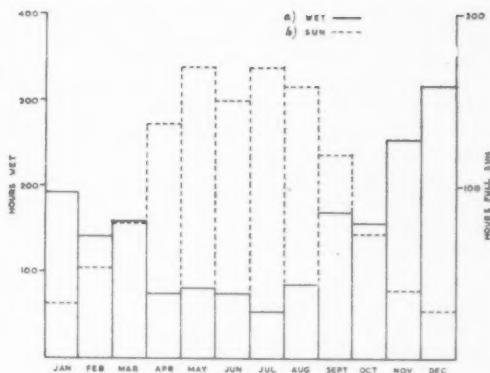


Fig. 2.—Monthly totals of periods for which painted surfaces were (a) wet, and (b) exposed to full sunshine. (Average for 1952-5). (T. R. Bullett<sup>(11)</sup>).

#### The Solar Spectrum

The quality of sunlight reaching the surface of the earth is variable from locality to locality and in the same region from season to season. The spectral composition will depend upon the amount of water vapour in the atmosphere, the number and nature of air-borne particles, the height of the atmosphere and many other meteorological factors.

P. Moon<sup>(9)</sup> has calculated a series of five curves to represent a standard mean spectral quality of sunlight falling on the earth's surface at sea level between zenith sun and zenith solar distance of 78.5 deg. Thus, except for high arctic latitudes, these calculated results cover all the daylight hours (Fig. 1).

#### (2) Temperature

The temperature which may be reached by a paint film in the course of natural weathering will vary greatly with the location and the range extends on either side of the maximum and minimum air temperatures. Metal panels painted in dark colours, exposed to tropical sunlight, may reach temperatures in the region of 100°C. On clear nights the loss of heat by radiation will reduce the temperature of exposed panels materially below air temperature. The rapidity with which these changes occur and the consequent thermal shock imposed upon the paint film will vary from season to season and place to place. In England the season most likely to produce severe thermal shock and disruption of an already weakened film is the spring and early summer. In the late summer even the South of England has over 16 hours between sunrise and sunset which minimises rapid temperature changes, and the almost constant cloudiness of autumn and early spring has the

same effect. In a similar way, the shortness of the winter day prevents a marked difference in temperature between night and day.

Thermal shock may be only a secondary factor in breakdown, in that it will have little or no effect until the film has reached a certain stage of fragility from other causes. The major role played by high temperature is to increase the rate of chemical action between the film and oxygen of the air and to increase the mobility of plasticizers and other liquid constituents held in it.

Table I

Variations in August-Florida			
Year	Hours Sun	In. Rain	Hours Wet
1941	239	3.12	198
1943	245	13.45	152
1945	298	5.30	65
1947	175	7.00	52
1949	204	6.76	47

Beckwith<sup>(10)</sup> reports some results of exposure tests at Florida by R. Wirshing. Identical panels were exposed at sites on the sea shore, four miles inland and eight miles inland. The panels on the waterfront and at the eight mile site were free from extensive cracking, those in the intermediate position suffered severely from this defect. His explanation of the difference in behaviour is that the prevailing East wind carried moisture over the first site without cloud formation, the air stream was forced upwards by the hot currents over the city of Miami, with formation of small clouds giving intermittent sun and shade with occasional showers in the latter part of the day. The furthest inland site was uniformly overcast with steady rain. The element of thermal shock was, therefore, markedly less at sites one and three than at site two.

### (3) Water in Contact with the Paint Film

Of all the factors in the natural weathering of paints, this is the most variable in respect of the length of time the film remains wet and the quality of the water falling on it. T. R. Bullett<sup>(11)</sup> has given an analysis of the weather at the Paint Research Station, Teddington, Middlesex, England. This is a small town in the Thames Valley on the Western or windward side of London (Fig. 2).

He sums up the conditions "Expressed as annual totals, full sunshine at Teddington for about 1200 hours, of which 900 hours is in the period April-September and only about 150 hours

Table II

Year	Number of Weeks Exposure							
	1	2	3	4	5	6	7	8
1948	86	85	62	38	23	10	—	—
1949	90	88	87	85	82	80	76	70

in the period November-February. Paint films are wet for about 1800 hours in a year, including roughly 500 hours in the period April-September and 900 hours in the period November-February. Thus, in winter, films are wet for about 31 per cent and exposed to sunshine for only about 5 per cent of the time; in summer the corresponding figures are 7 per cent and 22 per cent . . . Summing up . . . the conditions which are likely to develop visible breakdown occur most frequently in the same period of the year."

Rainfall varies over the surface of the earth from practically zero precipitation to 400 inches per year. Even in England over a distance of less than 200 miles the rainfall differs by a factor of 10.

A similar unpredictable variation in climatic conditions from year to year was reported by N. P. Beckwith for an exposure site in Florida (see Table I).

This variation was reflected in the results obtained in exposure tests. Two sets of panels coated with the same lacquer were exposed at an interval of one year, one in 1948 and the other in 1949. The first set chalked rapidly, whereas the second set showed a normal pattern of behaviour. Table II shows the gloss-meter readings on the two sets of weekly intervals.

Table III

Year	Hours Wet With Rain	Hours Wet With Dew	Total Hours of Sun
1948	23	123	344
1949	86	60	374

The climatic conditions during the period of exposure are summarized in Table III.

The enormous influence of contact with water is clearly shown by these figures. The amount of sunshine falling on the two sets of panels differed only by some 10 per cent, whereas the period for which the bad panels were wet with dew was double that of the good. Beckwith also records another confirmatory experience.

The effect of water in natural weathering depends too on stage in the life of the film at which it becomes saturated and simultaneously exposed to ultraviolet radiation. Thus, N. R. Fisk<sup>(12)</sup> states that panels first exposed in the summer differ in behaviour from the same system first exposed in the winter. He offers a similar analysis of the periods for which panels are wet to that given by Bullett<sup>(11)</sup>.

### (4) Atmospheric Contaminants

The only substance that can be considered as a non-factitious atmospheric contaminant is sea spray which may be carried great distances inland

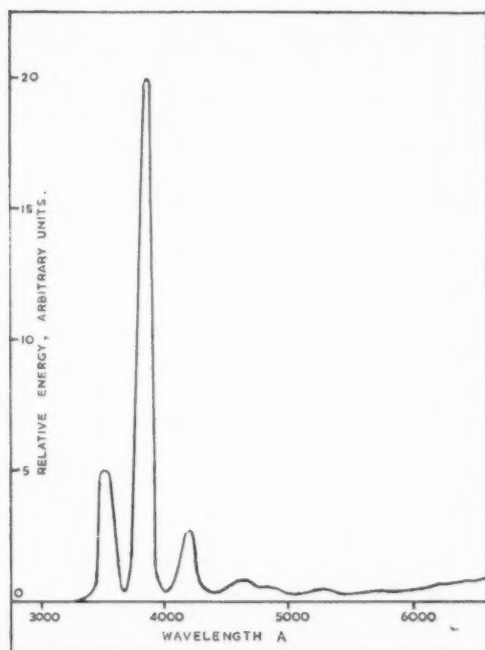


Fig. 3.—Distribution of energy in the spectrum of an enclosed-flame arc running at 12.5 amp., 130 volts, a.c. (Cooper and Hawkins<sup>(13)</sup>).

on the wind. The atmosphere over industrial cities, however, will contain appreciable amounts of sulphur dioxide and particulate matter derived from the combustion of coal, coke and fuel oil. The overall effect of all of these in the acceleration of actual film breakdown is probably slight, except in freak weather conditions such as the London "smog" of 1954, when the city lay under a black sulphur-laden blanket for days on end, or in the conditions existing in railway usage. The main cause of film disruption by sulphur dioxide is the passage of the gas through a wet film and subsequent attack upon the substrate whose corrosion products force off the organic finish.

#### Artificial Sources of Radiation

The sources of radiation used in accelerated weathering consist, in the main, of carbon arc lamps and mercury vapour discharge lamps. The carbon arcs are of the flame type and may be burned either in the open air or enclosed in a Pyrex globe fitting closely to the bed plate of the lamp so as to exclude all but a slow leakage of oxygen. The enclosed arc is the more convenient of the two in practice as it will burn for much longer periods without attention or re-trimming. High intensity carbon arcs require too frequent attention to be of great practical use.

The curves deduced by P. Moon<sup>(9)</sup> for the spectral value of sunlight have been shown in Fig. 1 and it is with these that the spectral distribution of the artificial sources must be compared. A study of the spectra of various lamps was made by Cooper and Hawkins.<sup>(13)</sup>

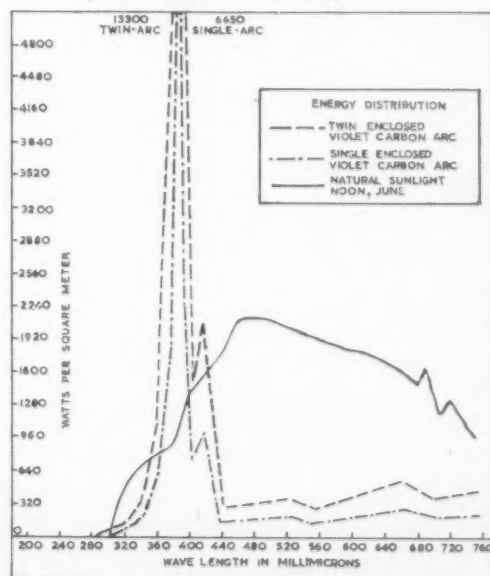
#### Enclosed-Flame Arc

The emission curve Fig. 3 is for an enclosed-flame arc running on alternating current consuming 12.5 amperes and with a voltage of 130 volts across the arc. This corresponds very closely with the condition of operation of the lamps used in a number of commercially available accelerated weathering units on both sides of the Atlantic. The effects produced by quite wide changes in electrical loading were negligible as was a change from alternating to direct-current operation.

The effect of the Pyrex globe, normally 2 mm. or less in thickness, was studied by these authors as well as that of the white powder which is deposited on the globe, after a short time of burning. Compared with the open arc, the clear globe had a negligible effect on the spectral distribution in the visible region, transmitting freely down to a wavelength of 350 m $\mu$ . After 16 hours burning, a fumed globe was still transmitting 90 per cent of the radiation transmitted by the clear globe with no preferential absorption.

The published transmission spectrum of an enclosed flame arc used in a commercial weathering

Fig. 4.—Energy distribution, twin and single and closed violet carbon arc, and natural sunlight, June. (Atlas Electrical Devices Co.).



apparatus, together with that given by makers as June noon sunlight, is shown in Fig. 4.

The general similarity between these curves, those of P. Moon<sup>(9)</sup> for sunlight and Cooper and Hawkins<sup>(13)</sup> for the enclosed carbon arc is immediately apparent and provides evidence that they are representative of the generality of enclosed arcs used for accelerated weathering. The claim that these arcs provide a spectrum "similar to that of noon June sunlight" has to be accepted with some reserve. The important lower wavelength end of the spectrum cuts off, it is true, at some 300  $m\mu$  in both cases but after that the similarity ends. Sunlight provides a continuous spectrum of substantially even intensity between 460  $m\mu$  and 690  $m\mu$  with a slight peak at 480  $m\mu$ . The arc spectrum has the bulk of its energy concentrated in a peak at 370  $m\mu$ .

### Mercury-Vapour Arc

The emission from an arc struck in mercury vapour contained in a quartz envelope in a convenient source of high-intensity radiation which has been used since the early work of Nelson<sup>(4)</sup> for the irradiation of artificial weathering specimens (see Fig. 5).

Two very significant differences are immediately noticeable between this spectrum and that of either the flame arc or of sunlight. The mercury-arc spectrum is discontinuous, most of the energy being concentrated in a few wavelengths, and there is an appreciable emission as low as 235  $m\mu$  compared with the cut-off wavelength of 300  $m\mu$  of both the arc and sunlight. In all, some 55 per cent of the emission energy is concentrated in the ultraviolet region, the remainder is in the infrared and visible spectrum, the visible light is concentrated in the three characteristic mercury lines.

### Xenon Arc

A newcomer to the field of lamps for accelerated weathering is the Xenotest arc produced by Quartz-lampen Gesellschaft mbH of Hanau. The arc consumes 1500 watts and is surrounded by a water jacket which absorbs all radiation of wavelength greater than 1.4  $\mu$ . It would therefore seem that, if this arc is employed in a weathering cycle, additional heating lamps would have to be used as in the Nuffield-Hanovia equipment described below.

The spectrum of the xenon arc is shown in Fig. 6 which is derived from measurements made by the Physikalisch Technischen Bundanstalt, Braunschweig.

This shows a much closer correspondence with the solar spectrum than any of the other arcs, particularly in the important region 300-360  $m\mu$ .

There is no published work on the use of the

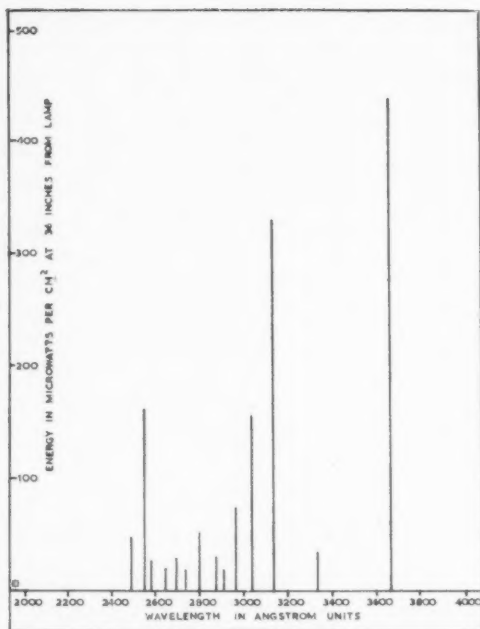


Fig. 5.—Energy distribution in the various lines of the spectrum of a typical medium-pressure quartz mercury arc. (Lamps Division, Englehard Industries Limited).

Xenon arc in artificial weathering of organic finishes, although some work has been done on the fading of dyestuffs, with very interesting results.

### Effects Produced by Varying Ultraviolet Sources

If the absorption spectrum of most paint vehicles is examined, it will be found that below a wavelength of about 300  $m\mu$  they are substantially "black," that is all wavelengths are absorbed with equal facility. Measurements of the quantum efficiency of the photo-oxidation of alkyd resins have indicated that the yield per quantum is independent of the wavelength between 250 and 320  $m\mu$ , that is to say that in this region all wavelengths are equally active (C. D. Miller<sup>(14)</sup>).

The shorter wavelengths penetrate less deeply into the film than the longer and so act more intensely in a limited surface layer and accentuate purely surface failures. A J. Birch<sup>(15)</sup> finds that the use of a mercury vapour arc under conditions of low relative humidity produces excessive micro checking on both alkyd resins and cellulose motor finishes, as compared to tropical exposure. The work of Nelson<sup>(4)</sup> using a mercury arc not only pointed in this direction but indicated quite clearly that ultraviolet radiation, on a completely dry

panel, produced very little effect beyond exaggerated fading of dyestuff pigment, coupled with a loss of gloss. The anomalous fading of pigments under ultraviolet light is frequently commented on in the literature on accelerated weathering.

The uniform nature of the ultraviolet absorption curve of most paint media is the probable reason why workers using sources of artificial radiation as different as the mercury discharge lamp, for example, H. A. Nelson,<sup>(4)</sup> A. J. Birch<sup>(15)</sup> and R. J. Brown<sup>(16)</sup> and those, like Dunkley and Fancutt<sup>(5)</sup> using the carbon arc, are both able to claim a high degree of correlation between their results and natural weathering for a wide range of paints. It is however necessary, when testing unknown materials, to be satisfied that selective absorption in a spectral region of the lamp that is not represented in sunlight does not occur. Apart from this, the literature supplies no reliable evidence of any differential effect, except that produced by intensity differences between various lamps. High-intensity lamps may produce unwanted side effects, not by their actinic radiation, but by an undue rise in temperature. Despite popular belief, ultraviolet radiation alone is not a potent agent of film degradation and increase of power of the arc produces much less than a proportional increase of physical failure by checking or cracking, although chalking is often unnaturally enhanced. Some measurements have been made of the quantum efficiency of the photochemical degradation of alkyd resins by E. B. Fitzgerald<sup>(17)</sup> who finds there is no evidence of any chain reactions and a low quantum efficiency of  $1 \times 10^{-4}$  for a range of wavelengths between 150 and 320 m $\mu$ .

### Moisture in Accelerated Weathering

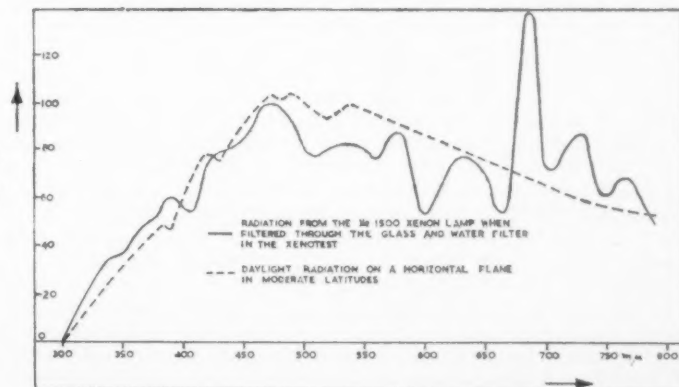
Outside industrial areas, where rain and dew may become severely contaminated with acid gases and "smog" particles, the water falling on naturally exposed paintwork will generally contain little in

solution except atmospheric gases, particularly carbon dioxide and oxygen. There is conflicting evidence as to the ability of ions to migrate through a paint film; R. S. Morell<sup>(18)</sup> could not detect the passage of ions, while Kittelberger and Elm<sup>(19)</sup> measured the transport of sodium ions through paint films and J. E. O. Mayne<sup>(20)</sup> found that polystyrene, linseed oil and coumarone-tung oil varnishes were selectively permeable to cations. Traces of heavy metal in water in contact with paint films can either promote or retard chalking, and copper is liable to give rise to staining. For these reasons, modern accelerated weathering equipment uses distilled, condensate or demineralized water conveyed in resistance glass or polythene pipes. Recirculation of the water is most undesirable as it quickly becomes contaminated with products leached out of the film and after contact with nitro cellulose lacquers the pH may fall sharply.

The effect of water cannot be considered on its own as it is the combination of water and irradiation which produces an amount of film degradation, much greater than the combined effect of each agent applied separately. Together they are the most destructive agents, exceeding in potency high temperature and industrial atmospheric contaminants.

It is important, if the mode of failure observed in the field is to be reproduced in accelerated cycles, that the natural balance between the effect of water and of irradiation per se is not upset. T. R. Bullett<sup>(11)</sup> suggests the weather analysis made at the Paint Research Station, Teddington, Fig. 2, indicates that for one third of the time naturally weathered paint films are neither wet nor exposed to sunlight and that for one sixth of the wet period there is no irradiation. Continuous wetting and radiation at level no higher than summer sun intensities would therefore alone give a fourfold acceleration without subjecting the film to a disruptive agent at a higher level than occurs in nature.

Fig. 6.—Xenon-arc spectrum and combined sky and solar spectrum in medium latitudes. (Quartz-lampen, G.m.b.H.).



The exact ratio between wet and dry film irradiation will vary from cycle to cycle and depend upon such factors as the temperature reached by the panels, the humidity maintained within the apparatus and disposition and frequency of the water sprays. The amount of wet irradiation tends to increase as more experimental data is accumulated. Thus Nelson<sup>(4)</sup> began with entirely separate exposures to mercury-arc radiation and water and, over the years, built up the wet irradiation time until H. A. Nelson, F. C. Smutz, D. L. Gamble<sup>(21)</sup> incorporated some period of irradiation at 100 per cent humidity for a time based on local conditions. A. J. Birch<sup>(15)</sup> has obtained better correlation in a mercury arc apparatus by increasing the amount of spraying and raising the prevailing humidity. At lower humidities micro checking had been excessive.

The ratio of wet to dry irradiation and the humidity in the test chamber must all be controlled, as should the pH of the water used. The cycle to be used in different cases can only be arrived at by trial and error as it will depend upon the materials being tested and the type of climate it is required to simulate. The importance of the dew and humidity conditions and their relation to irradiation cannot be overstressed, since they remain perhaps the only genuine variables in the design of weathering cycles since the quality of the radiation appears not to be critical.

### Temperature

The effects of high temperature in accelerated weathering cycles are parallel with those produced by high temperature in natural weathering. That is to say the effect varies from composition to composition, being greatest for solution type coatings and oil paints, and least for stoving or polymerised finishes. It is, therefore, a legitimate deduction that any attempt to accelerate film failure by raising temperature will increase the discordance observed between accelerated weathering tests on different materials. The rate of acceleration of film failure for an oil paint produced by a rise in temperature above normal will be greater than for an alkyd and greater still than for an epoxy or amine resin film. High temperatures sometimes produced by the use of powerful arcs can slow down film deterioration by stoving the paint and reducing the loss by erosion. Case hardening of thick films can cause premature cracking. Most accelerated cycles control the panel temperature somewhere in the region of 50-60°C. The desirability of so doing has been made mandatory by the American Society for Testing Materials.

Refrigeration of accelerated weathering specimens has been incorporated in a number of cycles, but its utility seems doubtful. As the use of a refrigeration period can only be obtained by inter-

rupting the general sequence and transporting the panels to a suitable cold room, it has tended to drop out of modern cycles.

### British Accelerated Weathering Cycles

There are three accelerated weathering cycles in Great Britain which have become part of specifications and are well documented.

#### (a) British Railways

This was described by Dunkley and Fancutt<sup>(5)</sup> in 1953 and had then been in use for nearly 20 years to assess the quality of paint submitted for use on railway rolling stock, equipment and buildings. Two cycles were employed according to the intended use of the paint. The basis of both cycles was exposure to ultraviolet light and water. Panels are mounted vertically on a horizontal drum. At either end of a diameter is mounted an enclosed alternating-current arc at seven inches from the panels. A second diameter at right-angles carries a pair of sprays. The drum rotates at three revolutions per hour. The arc consumes 13 amp. at 125-135 volts.

The programme of exposure constituting Cycle 1 is :—

17 hour lamps and spray } Continued for 5 days  
6 hour lamps only } followed by 2 days rest.

Table IV

		Cycle One per cent	Cycle Two per cent
Bituminous paints	Full agreement	25	66
	Partial agreement	44	7
	Disagreement	31	27
Oleo resin varnishes	Full agreement	66	72
	Partial agreement	25	23
	Disagreement	9	5
Synthetic resin (alkyd) varnishes	Full agreement	75	71
	Partial agreement	20	29
	Disagreement	5	0

Full agreement = Order of merit and type of failure similar.  
Partial agreement = Order of merit different type of failure similar.  
Disagreement = Neither order of merit nor type of failure similar.  
Extract of correlation between natural and accelerated weathering (Dunkley and Fancutt<sup>(5)</sup>).

Cycle 2 uses the same apparatus as Cycle 1 with the addition of exposure to sulphur dioxide in a separate chamber. The gas is passed into a saturated solution at the bottom of the exposure vessel at a rate of 1-2 bubbles per second, the atmosphere temperature is held at 35-40°C. and 100 per cent relative humidity. The duration of exposure to this atmosphere is two periods of six hours each per week. Sulphur dioxide is passed in commencing 3 hours after the panels are placed in the chamber.

The correlation claimed varied with different types of materials and was in general better for Cycle 2 than Cycle 1, but the conditions of use of

paints tested in Cycle 2 were such as to make sulphur dioxide the most destructive agent.

The panels were analyzed for gloss, checking, chalking, colour stability, protection, appearance and adhesion.

#### (b) Paint Research Station

This apparatus was developed by the Paint Research Station by P. J. Gay<sup>(22)</sup> and adopted by the Ministry of Supply. It consists of a 48-inch diameter drum round the inside of which three tiers of specimens can be arranged in galleries. The source of radiation is an enclosed direct current carbon arc consuming 850-950 watts at 110 volts suspended 8-9 inches from the centre of the drum. Three atomizers spraying one Imperial gallon each of water per hour are set vertically at the centre of the drum, one opposite the centre of each tier of panels. The drum revolves once every 20 minutes and is fitted with a lid which does not revolve with it. The atomizers are so situated that the panels are sprayed for a quarter of a revolution before they approach most closely to the arc. The temperature inside the drum is measured by a thermometer projecting through the lid and regulated to read between 38 and 44°C. by raising or lowering the lid. The light and sprays are operated continuously, thirty minutes are allowed in each twenty-four hours to change the carbons. Panels are examined every forty-eight hours for fading, chalking, loss of gloss, cracking, blistering and checking.

No correlation figures with outdoor exposure are given.

#### (c) The Nuffield-Hanovia Accelerated Weathering Unit

This is based on work in the Nuffield Central Research Laboratories by Birch and Brown<sup>(15,16)</sup> and it has formed the subject of a number of papers read to the Organic Finishing Group of the Institute of Metal Finishing, but which have not appeared in the Transactions. The apparatus consists of a circular ring 47 inches in diameter around which sample panels are suspended by means of clips. The ring rotates once an hour. At the centre of the ring is a vertically mounted water-cooled mercury-arc lamp consuming 3.8-4.0 amp. at 280-300 volts with constant wattage control. The spectrum of the lamp has been shown in Fig. 4. The water in the lamp jacket is maintained at 66°C. The panels are heated by 24 infrared lamps of 250 watts each and the panel temperature is indicated by a mercury thermometer fastened in contact with a black painted panel. When this has reached the desired temperature, which varies at different parts of the cycle, the two thermostats controlling the infrared lamps are set. Water spray is by two Aerograph Type FN-504 stainless-steel atomizing type guns. Distilled water

conveyed from a stainless-steel tank by polythene piping, is used. The remarkable things about this cycle are its use of independent heating, controlled variation of temperature at different parts of the cycle, a high-intensity mercury arc and the elaborate nature of the controls.

The cycle employed is :

- 9 a.m. to 12 noon Irradiation, two water sprays in operation. Black panel temperature 140°F.
- 12 noon to 2 p.m. Irradiation without water. Black panel temperature 180°F.
- 2 p.m. to 5 p.m. Repeat first three hours.
- 5 p.m. to 9 a.m. Shut down.

The work done with the machine has been in the main on automobile finishes and from the analysis of 3200 panels, A. J. Birch<sup>(15)</sup> claims accurate reproduction of tropical failure is possible in 10 days exposure. Comparisons have been made with a number of stations but the majority with panels exposed at Singapore, which is regarded as average, and at Colombo, which is considered severe. The criteria are fading, loss of gloss, cracking and checking, which are assessed visually with the aid of a binocular microscope.

### Conclusions

The question before all who have to rely on accelerated weathering cycles is how far can they be relied on? The work which has been studied makes it appear inherently improbable that any cycle can be derived to reproduce failure under all kinds of climatic conditions or even to rate all paints in the correct order of merit when exposed to a single climatic environment of a normal degree of variation. This weakness of accelerated weathering has been recognised most strongly by those who have carried out the most valuable and painstaking work in the subject. Dunkley and Fancutt<sup>(5)</sup> though they claim 70 per cent correlation (Table IV), lay down the rule that accelerated weathering must be employed only in conjunction with all the usual chemical and physical tests and samples must be tested using materials of similar composition as controls. They warn against attempting to estimate the life of paint under service conditions from accelerated weathering results.

Table V

	Singapore per cent	Colombo per cent
Alkyd synthetics	98.3	32.5
Cellulose	93.1	94.1

Correlation between exposure at Singapore and Colombo and the Accelerated Cycle, percentage complete agreement.

(Continued in page 138)

### The Accelerated Weathering of Organic Finishes.

(Continued from page 137)

P. J. Gay<sup>(22)</sup> also sets out the limitations which he has found in the use of accelerated weathering cycles, which he lists as—

- (a) "Failure . . . to predict the mode of breakdown, a point upon which information may be of great importance.
  - (b) The very slow rate of breakdown observed with many dark coloured gloss paints—a rate which sometimes has little relation to their external durability.
- Further, quite misleading results are obtained in a small proportion of cases, particularly where rather thermoplastic films are being tested."

He suggests that when the results are interpreted with caution and due regard to the limitations of the technique, results of value and significance can be obtained as a rough sorting test for order of merit of a range of paints and "to assess the durability of a range of compositions of similar type where considerable experience exists as to the general behaviour of such compositions on external exposure." In this case the results can be accepted with greater confidence and small differences may have greater significance.

The analyses of weather which have been given in Fig. 1 and Tables I and III show a high variability from year to year in a single place. Many industrial paints may be exposed in service all over the world and in the case of motor cars move frequently from one environment to another. The idea of the durability of a paint is of necessity a purely subjective synthesis which is made with time and experience, not only from exposure panels which have limitations, but from actual service records obtained from painted goods and structures. Until a similar bulk of information, often incommunicable, has been built up for accelerated exposures and experience has shown the shortcomings and strength of the particular system used, interpretation will be unreliable. The method of procedure which has found greatest favour is to select the most aggressive climate to which the paint will be subjected and to try, by adjusting the cycle, to obtain good correlation with it in the hope that all apparently less severe climates will be included. This is often not the case as widely differing environments may produce failures so different in kind as not to be included in the selected cycle. It is still as a sorting device among known compositions that accelerated weathering has proved to be most useful. It is perhaps most significant that no paint manufacturer, as far as the author is aware, will market a product on the

strength of accelerated weathering alone but continues to rely upon natural exposure data for the final verdict.

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### "SAFETY AND SOLVENTS."

A NEW 16-mm. colour film on the safety precautions required in the handling of highly flammable solvents is available from the publicity department, chemical division, Distillers Co. Ltd., Devonshire House, Mayfair Place, London, W.1.

This film was made at the Salt End, Hull industrial chemicals factory of D.C.L. Chemical Division and the elaborate precautions taken at a chemical factory, particularly those designed to prevent the generation of static electricity, are detailed with the assistance of animated diagrams.

The D.C.L. factory at Hull obtains the greater part of its raw materials by sea tanker from the Grangemouth plant of British Hydrocarbon Chemicals Ltd. The film shows plants at the Grangemouth site which produce these raw materials. At Hull, the precautions taken from the point of arrival of a sea tanker at the Salt End jetty and the discharge of the load by pipeline to bulk storage are shown together with a typical modern process plant, intermediate product storage, final product storage and the procedure at bulk and drum filling installations.

Fire-fighting in a chemical factory calls for special techniques. The film includes a realistic turn-out by both the works brigade and East Hull, whose co-operation is gratefully acknowledged.

# MULTILAYER METAL-CERAMIC COATINGS

By  
W. L. AVES, Jr.\*

## for High-Temperature Protection

### Introduction

**A**IRCRAFT and industrial requirements during the last few years have necessitated coatings capable of protecting base materials at high temperatures under varying conditions of oxidation, erosion, heat flux, vibration, thermal cycling, and time of exposure. The purpose for which the described coating system was developed is to extend the life of the base material under both calculable and observed environmental conditions of service.

The scope of this article will be principally devoted to the application of multilayer refractory metal-oxide type ceramic coatings on graphite-base materials. No one coating system will suffice for use in protecting graphite under such diverse environmental conditions such as those encountered on solid propellant rocket nozzle inserts, and those on glide-type atmospheric re-entry leading edges. Screening, calculated environmental, and actual service tests are required before the optimum coating system can be selected.

Graphite has been used successfully for solid propellant rocket-nozzle inserts, jet guidance vanes, and linings in rocket motors as a result of its high sublimation point, resistance to thermal shock, ease of fabrication, and availability. However, its use is limited by its poor resistance to high-temperature oxidation and gaseous erosion.

In the past considerable work has been performed to provide graphite with integral metal-carbide coatings (*i.e.*, silicon carbide, chromium carbide, and zirconium carbide). Coatings of this type have extended the service life of graphite under high-temperature oxidizing conditions considerably. However, coatings of this type on large configured areas were unreliable in that flaws and discontinuities, some microscopic in size, frequently existed. Any flaw in the coating leaves the underlying graphite vulnerable to catastrophic oxidation. Lack

of a suitable non-destructive method for checking coatings of this type over large configured surfaces for imperfections has resulted in numerous application failures.

The general method of approach used in this investigation was to advance the state of the art of high-temperature coating systems. Coating development and evaluation work led to the use of unique metal-ceramic laminated coatings in several high-temperature problem areas.

### History of Development

Initially, it was necessary to develop a high-temperature coating system to provide aircraft safety in the event of an inadvertent rocket motor ignition. In this case it was essential to contain a 4,500°F. (2482°C) rocket motor blast under full burn out conditions.

A test breech was subject to a hang-fire test. In approximately 0.7 seconds, the breech dome burned through when subjected to the exhaust of a rocket motor. This dome was constructed from 0.125-inch 17-7PH stainless steel, heat treated to 190,000 lb. per sq. in. ultimate tensile strength and coated with a flame sprayed 0.010-inch thick alumina coating. Essential deflection of such a blast at right-angles to its direction of flow, under full burnout conditions, resulted in the development of the laminated metal-ceramic coating system.

When the existing factors of space limitation, weight, shape and fabrication were taken into consideration, only one approach to solve the problem appeared practical. It was necessary to provide a protective coating or liner to the inside face of the breech dome.

The breech was reworked to evaluate replaceable test domes and mounted to accept rocket motors. The fins of the rockets were removed and the rockets held in the tube with a bar across their nose to permit full burn-out of the rocket motor against the test surfaces of the replaceable test domes. The test plates were fabricated by spinning 0.071-inch

\* Chance Vought Aircraft, Inc., U.S.A.

thick 17-7PH steel and heat treating to 190,000 lb. per sq. in. ultimate tensile strength.

The failures obtained from the initial tests stimulated a new line of thought which resulted in the development of the multilayer coatings. Three multilayer molybdenum-aluminum oxide coated test domes were partially sandblasted after test to present a cross-section of the initial 0.050-inch coating. Subsequent tests proved a 0.050-inch laminated coating adequate to protect even a 0.060-inch aluminum or a 0.060-inch thick glass-fibre back-up dome.

The promising results obtained from the blast deflector application led to the extended development and use of the coating to protect a fixed afterburner pressure rake. This coating is capable of protecting the base material to 3,000°F. in the cone of the afterburner of a J-57 or J-75 jet engine. Service conditions incur high thermal shock, severe gaseous erosion, and high acoustic levels (150 decibels) for up to 30-minutes' operation in full afterburner for a specified number of cyclic conditions.

#### Multilayer Coating for a Graphite Rocket Nozzle Insert

Recently, it became necessary to develop and evaluate a protective coating for a rocket nozzle and graphite throat insert. The nozzle shape chosen for the solid propellant rocket was the conventional convergent-divergent conical nozzle using a 15-degree half-cone angle. Expansion ratios of 6 to 1 are evolved from the use of this system, resulting in a chamber pressure of 1,000 lb. per sq. in. The nozzle weight, insert design, and protective coatings were based on a 35-second burning time. The major problems encountered were those of keeping the nozzle temperatures at an acceptable level, and to minimize erosion in the throat.

To ensure that no failures occurred due to heat feedback from the nozzle, a layer of zirconia ( $ZrO_2$ ) material between the graphite insert and the steel structure was incorporated. The graphite throat insert and steel exit cone were coated with a multilayer coating of molybdenum and aluminum oxide. Initial operative tests using such single-layer coatings as aluminum oxide, zirconium oxide, and molybdenum resulted in failure. The oxide coatings would not withstand the erosive conditions existent, and the molybdenum coatings unreliably separated at the molybdenum-to-graphite interface on thermal shock and entered the exhaust gas stream resulting in severe erosion of the insert throat.

Static testing of the rocket motors showed that careful attention to the nozzle surface finish and the inlet conditions greatly affected the erosion of the insert. The first three nozzles produced, had

imperfections resulting from the application technique that was employed. In all three cases, erosion of the coating and graphite insert occurred during test. When the method of manufacture of the coated nozzle had been perfected, almost perfect surface contours were obtained with a 64 r.m.s., or better, surface finish. The nozzle throat diameter changed little, if any, during test on motors 4, 5, 6, and 7 (Table I). Subsequent flight tests with this rocket have proved this coating to be 100 per cent reliable for this specific environment.

**Table I**

Ballistic Parameters

Motor	No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7
Burning time in seconds	20.80	19.36	18.35	17.60	22.70	27.00	20.00
Initial throat diameter inches	1.602	1.598	1.590	1.600	1.595	1.600	1.596
Final throat diameter after firing, inches	1.624	1.610		1.60	1.601	1.60	1.596

Average specific impulse varied from 195-212 lb.-sec./lb.

Average thrusts varied from 1,027 to 1,305 lb.

Stagnation temperature in throat calculated to be 4,600°F.

Exit diameter of nozzle 3.61 in.

The propellant selected for this motor was a castable, polyurethane-base formulation containing ammonium perchlorate as the oxidizer.

#### Coating Application Technique Employed on Rocket Nozzles

After machining a suitable nozzle-grade graphite to allow for coating build-up, the insert was chucked in a lathe. Surface roughening was accomplished by use of a wire brush incorporating 0.010-inch round-tip stainless-steel bristles. As the lathe turned (20 r.p.m.), the brush was inserted and a light (3 to 8 lb.) pressure was applied as the brush was pushed across the surface of the insert for about ten revolutions. The loose graphite was then removed by use of a compressed dry air blast across the surface of the insert.

A thin 0.002-inch coating of molybdenum was then applied by use of a conventional oxyacetylene metallizing gun. This was followed by the application of a 0.006-inch coating of aluminum oxide applied from rod form by feeding a cylindrical extruded alumina rod, 1/8-inch diameter, at 6 inches per minute, into the tip of an oxy-acetylene flame. Compressed air at 80 lb. per sq. in. projected the plastic particles about 4 inches to the molybdenum where they flattened and froze. Another 0.002-inch coating of molybdenum was then applied followed by a 0.006-inch coating of alumina, and a final coating of molybdenum about 0.004-inches in thickness. This resulted in a total coat thickness of 0.020 inches before finish grinding. The insert throat is then ground to size by the removal of approximately 0.002 inches of the final

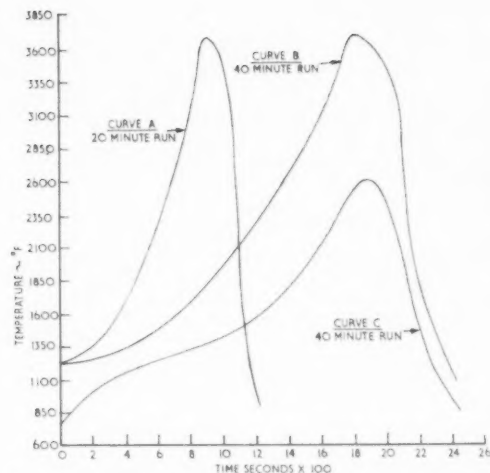


Fig. 1.—Approximate time-temperature profiles for flame-test screening.

coating of molybdenum leaving a total coating thickness of 0.018 inches. The finish grinding operation sizes the throat, and provides the necessary surface finish for optimizing its efficiency during operation.

Additional development and evaluation work on high-temperature coatings at CVA is primarily directed towards two widely varying high temperature environmental conditions. One entails a coating capable of protecting graphite at very high temperatures (up to 3,076°C.) in a severely erosive high-velocity gas stream for time periods up to 40 seconds in a reducing environment. The other condition necessitates a coating capable of protecting graphite when exposed to aero-dynamic heating approaching 2204°C. for prolonged time periods, in an oxidizing environment.

The environments to which the metal-ceramic coatings were exposed in the discussion so far were all reducing.

Reliable protection for graphite leading edges exposed to the environmental conditions expected during glide type re-entry, present an altogether different problem than those previously discussed. Aerodynamic heating to above 1650°C. is encountered in an oxidizing atmosphere under this condition. The leading edge must be capable of reliably withstanding oxidation at temperatures near 1926°C., erosion, thermal shock, high noise levels (160 decibels) and thermal cycling. Ability to withstand thermal cycling during such a flight is necessitated by the moderate temperature peak during boost, and the higher peak during re-entry.

A coating capable of protecting such a leading edge is required to have the following properties:

1. High-temperature oxidation resistance.

2. A spectral emissivity above 0.7 at 1926°C.
3. Resistance to high heat flux.
4. Thermal expansion compatible with the base graphite.
5. Erosion resistance.
6. Reliable adherence.

Additional requirements are reproducibility, and a reliable technique for application that will not detrimentally effect the base graphite.

Considerable testing and development work was required before a reliable coating system could be developed that would protect graphite under the above parameters. A description and the results of some of these tests are presented.

### Screening Tests

#### Oxyacetylene Torch Test

The purpose of this test is to determine the ability of the coatings under consideration to withstand pre-determined time-temperature variations similar to those expected in service. Some time-temperature profiles expected to be encountered in a hypersonic environment are shown in Fig. 1.

The torch test facility is set up as shown in Fig. 2. The operator moves the torch to and from the specimen on a manual crank in order to follow a predetermined time-temperature profile. The temperatures are measured by use of both an optical and radiation pyrometer. The observed temperature is corrected for emissivity by using the relative emissivity determined during the test.

The relative emissivity was obtained by using correction curves with the optical and radiation pyrometers. The procedure for doing this is given in WADC Technical Report 57-577, Part I (AD150957).

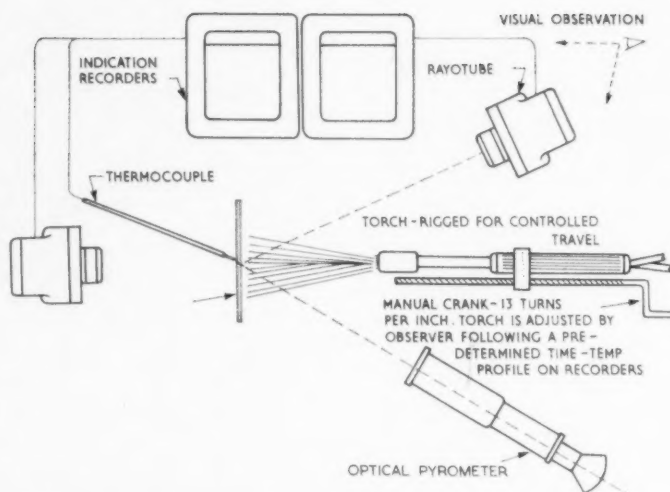
Table II gives the results of a few informative torch tests on coated graphite base materials. All of the samples tested in Table II were 4 in. x 4 in. x 1/4 in. dimension.

The ability of a laminated metal-ceramic coating to reduce the heat flux imposed on the base material is evidenced in tests 8 and 9, Table II. The siliconized and nitrided graphite apparently failed due to thermal shock in test 8. In test 9, a multi-layer coating reliably corrected this drawback to the use of siliconized and nitrided graphite.

The results of testing numerous coating graphite samples are presented as follows:

1. Siliconized graphite presented a reliable coating base.
2. Chromia, because of its tendencies to blister, was suitable for use only as the final coating to increase emissivity.
3. The optimum coat thickness appeared to be about 0.030 in. for the protection of graphite.
4. Metal oxide layer thicknesses in excess of 0.008 in. lack bond strength during thermal cycling.

Fig. 2.—The thermal test set up, showing the manual crank used to move the torch to and from the specimen in order to follow a predetermined time-temperature profile.



5. The best coating for siliconized graphite subjected to the time temperature profiles given in Fig. 2, consisted of three layers of 0.003 in. molybdenum applied alternately with 3 layers of 0.005 in. alumina and a final layer of 0.005 in. chromia.

As a result of the above screening test, the optimum coating system described in No. 5, above, was applied to a larger siliconized graphite dome and tested in a multiple torch test jig.

#### Oxidation Tests

This investigation is conducted in order to obtain oxidation resistance data on the coating

materials under consideration for use. A schematic diagram of the high temperature oxidation test facilities is shown in Fig. 3. The percentage oxidation of the protected base material was measured as a result of weight change and visual appearance of the test specimen after test.

In this test, coating efficiency is measured by its ability to prevent oxidation of the base material in a representative time-temperature-oxidation environment to that expected in service. Table III gives the results of a few representative oxidation tests on coated graphite materials. Specimen size is approximately 1/2 in. x 1/2 in. x 2 in.

Temperature measurements are made by use of

Table II

Results of a few Representative Oxyacetylene Torch Tests on Coated Graphite

Test No.	Base graphite	Coating System	Total exposure time	Peak temperature reached*	Curve	Resultant appearance
1	896G	1 layer 0.020 in. molybdenum	40 minutes	3,740° F.	B	Small blisters appeared in coating.
2	896G	Chromalloy W-2 coating 2 layers 0.004 in. molybdenum 2 layers 0.006 in. alumina 1 layer 0.004 in. chromia	40 minutes	3,550° F.	B	Light erosion Some surface coating blistering at 18 minutes. Adherence good
3	896G	1 layer 0.020 in. alumina	20 minutes	3,610° F.	A	Coating cracked and spalled on cooling. Adhesion was lost, some erosion of base occurred
4	3474	3 layers 0.003 in. molybdenum strips 3 layers 0.005 in. alumina 1 layer 0.004 in. chromia	40 minutes	3,550° F.	B	Some surface blisters
5	3499	5 layers 0.002 in. molybdenum strips 5 layers 0.005 in. alumina	40 minutes	3,550° F.	B	Unaffected
6	AUF	1 layer 0.020 in. molybdenum Chromizing Co. Durak MG	40 minutes	3,130° F.	B	Small blisters appeared. Otherwise unaffected
7	896G	5 layers 0.001 in. molybdenum 5 layers 0.003 in. chromia	20 minutes	3,770° F.	A	Many blisters appeared in coating
8	ATJ	Siliconized and nitrided	28 minutes	3,100° F.	B	Specimen cracked completely through and 1/3 distance across the face when reaching 3,100° F. at 27 minutes
9	ATJ	Siliconized and nitrided 3 layers 0.003 in. molybdenum 3 layers 0.005 in. alumina 1 layer 0.004 in. chromia	40 minutes	3,580° F.	B	Unaffected

\* 3000° F. = 1650° C. approx.

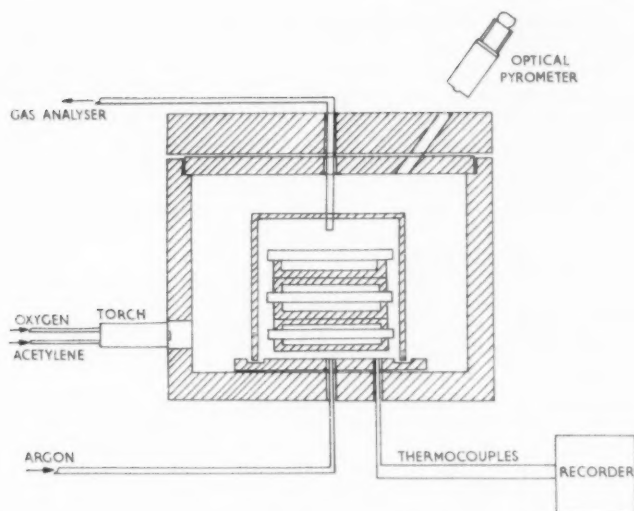


Fig. 3.—A schematic diagram of the high-temperature oxidation test facilities.

a platinum-platinum rhodium thermocouple, and an optical pyrometer for temperatures up to 2500°C. The percent oxygen is controlled by introducing argon and oxygen to the retort and setting control by use of an orsat analysis of the retort exhaust gas.

Results obtained from the oxidation tests presented the following summations:

1. A siliconized coating applied to molybdenum coated graphite offered good oxidation resistance.
2. A siliconized coating applied to molybdenum-alumina multilayer coated graphite provided good high temperature oxidation resistance.
3. In a molybdenum-ceramic coated graphite specimen, the molybdenum oxidized, yet it effectively protected the base graphite from severe oxidation.
4. The siliconized and nitrided graphite exhibited slight surface reaction with no visible cracks.

#### High temperature tensile tests

The purpose of these tests was to determine the ability of the coatings to adhere to the graphite, and to evaluate the effects of the coating and its application on the mechanical properties of the material under load, temperature and time conditions.

The tensile tests were performed at off-site testing facilities on a high speed tensile test machine. Heating was accomplished by using the specimen as an electrical resistance for the passage of electric current. The temperature of the specimens was measured by using thermocouples for temperatures up to 3,000°F. (1650°C.) and optical and radiation pyrometers for the higher temperatures.

The extensometer used was constructed of ceramic to afford electrical and thermal insulation. The extensometer arms translated the deformation within the gauge length of the specimen into the

Table III

Representative Results of Oxidation Studies on Multilayer Metal-Ceramic Coated Graphite.

Material	Coating	Total time hours	Max. <sup>3</sup> temp. °F.*	Weight diff. per cent	Per cent O <sub>2</sub>	Remarks
3499 <sup>1</sup>	4 layers 0.002 in. Moly	6	3500	- 3.0	12	Coating bloated and cracked
3499	4 layers 0.005 in. alumina	6	3500	- 0.07	12	Blistered and cracked along edges
3499	1 layer 0.020 in. molybdenum Chromalloy W-2 coating	6	3500	0.1	12	No apparent change
3499	3 layers 0.002 in. Moly					
3499	4 layers 0.005 in. alumina					
ATJ <sup>2</sup>	W-2 diffused coating	6	3500	-15.2	12	Severely oxidized
ATJ <sup>3</sup>	Bare	7	3800	- 1.7	11	Some small blisters appeared on surface
ATJ	Siliconized and nitrided					
ATJ	Siliconized and nitrided	7	3800	- 0.9	12	Some blisters appeared, otherwise unaffected
ATJ	3 layers 0.002 in. molybdenum					
ATJ	3 layers 0.004 in. zirconia					
ATJ	W-2 diffused coating					
ATJ	Bare	7	3800	-20.8	12	Severely oxidized

1—Average rate of climb 875°F. (460°C.) per hour. 2—Average rate of climb 750°F. per hour. 3—Time at maximum temperature 2 hours. \*3500°F = 1926°C. approx.

flexure of a spring, to which strain gauges are mounted and connected into a strain gauge bridge for strain measurement. Table IV shows some average results for metal-ceramic coated graphite tensile specimens. The coating in each case was identical. The coating applied was composed of three layers of 0.002 in. molybdenum applied alternately with three 0.005-in. layers of alumina to which a final coating of 0.005 in. chromia is applied.

**Table IV**

Representative Tensile Properties of Coated Graphite at Different Temperatures and Holding Times, and at a Nominal Strain rate of 0.01 in./in./sec.

All Specimens Heated to Test Temperature within 90 sec.				
Specimen number	Temp. °F.	Time at temp. Sec.	Ultimate str. 1,000 p.s.i.	Elong. per cent.
1	2000	1800	2.10	0
2	2800	1800	2.37	0
3	3000	1800	3.03	0
4	4000	10	2.75	0

Nominal specimen gauge diameter 0.375 in.; diameter including coating, 0.420 in.

Calculation of ultimate strength based on specimen diameter of 0.375 in.

Even though an extensometer was used to follow cross-head travel, there was virtually no plastic deformation of the graphite, even at 4,000°F. (2204°C.) therefore, no yield strength was obtained.

Results from these tests proved that neither processing technique or the applied coating had any detrimental effects on this physical property of the graphite. The coating provided adequate protection to the graphite under the conditions of test in the air environment

### Ram-Jet Test

The purpose of this test was to evaluate coated graphite under conditions considered more severe in many areas than those expected in a glide re-entry type of environment. Fig. 4 shows a diagram of the test facilities.

The supersonic ram jet burner is a Marquardt Aircraft Company Model AH2A heater utilizing compressed air, gaseous oxygen, and grade JP-4 aviation fuel which are mixed and burned in the unit, yielding a supersonic stream of high temperature combustible products. The jet is 8.1 inches in diameter at the nozzle exit. The jet properties are varied by changing the ratio of fuel to air, oxygen to air, total mass flow, or all three. A choice of mixture ratios and the total mass flow rate fixed the stagnation temperature and pressure. The ram motor is capable of operation at stagnation temperatures of approximately 4,000°F. (2204°C.) by the addition of gaseous oxygen. Under these conditions, the jet is oxidizing (up to 5 per cent excess oxygen).

A typical ram-jet test includes one in which the configured part under test was advanced toward the jet exhaust from the 120 inch starting station in 20 inch increments at 90 second intervals. From the last 20 inch station the part was normally advanced to the 2.7 inch station (tip stagnation point within the first mach diamond) and held for 5 minutes before back-off.

Results of this test on pre-selected promising coating systems have provided the following information:

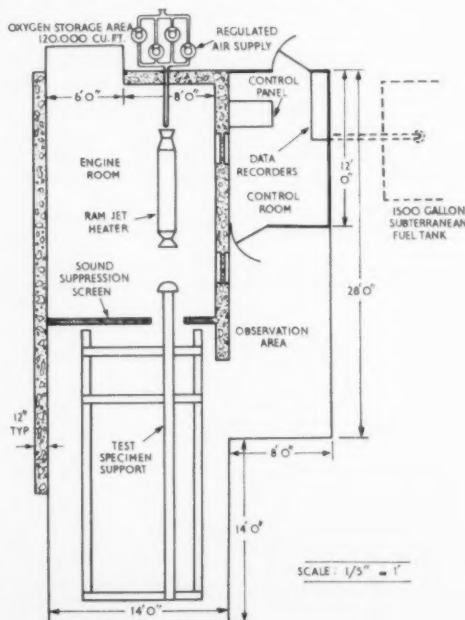
1. Diffusion coatings such as siliconized graphite and siliconized and nitrided graphite did not reliably protect the graphite from oxidation because of small pin hole type flaws and discontinuities in the coating.

2. The molybdenum - alumina - chromia and molybdenum-zirconia-chromia multilayer coating systems melted and flowed from the hottest section of the pre-siliconized graphite part, but not until they had suitably extended the life of the part to where it would pass the test.

3. Once through-flaw oxidation commenced on the the siliconized graphite it tended to balloon out under the siliconized skin rapidly with a resultant catastrophic failure.

(Continued in page 152)

Fig. 4.—Diagram of the ram-jet test arrangement.



# Some Recent Advances in ANODIZING PRACTICE AND KNOWLEDGE

By F. C. PORTER, M.A., A.I.M.\*

(One of a series of lectures, presented under the title of "Recent Developments in Electrolytic Metal Finishing" at the Borough Polytechnic, London, and published by permission of the Principal).

THE ever-increasing use of anodized aluminium, particularly in bright trim and in architectural applications, is due largely to the development both of special aluminium materials and of improved processing techniques. Other operational changes, notable automatic or continuous plants, stem from the need for speedier, simpler and more economic means of handling the growing quantities of work.

## Nature of the Anodic Film

The anodized surface on aluminium consists of alumina (aluminium oxide) with modifications derived from the electrolyte or from the alloying elements of the basis metal. The films formed in sulphuric acid contain 13 to 15 per cent. sulphate, those in phosphoric acid about 6 per cent. phosphate but those in chromic acid only about 0.2 per cent. chromate. Some water is chemically retained in the film at the sealed surface which corresponds mainly to the formula  $\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$ . The unsealed film is virtually anhydrous.

Heterogeneous alloys give rise to differing oxidation rates. Of the inter-metallic phases present in aluminium alloys, silicon-rich phases,  $\text{FeAl}_3$  and  $\text{MnAl}_6$  are not oxidized;  $\text{CuAl}_2$  dissolves slowly in sulphuric acid, while  $\text{Zn}_3\text{Al}_2$ ,  $\text{MgZn}_2$ ,  $\text{Mg}_2\text{Si}$  and  $\text{Al}_2\text{Mg}_3\text{Zn}_2$  in approximately that

order dissolve more rapidly. The final appearance can often be related to the colour and refractive index of the oxides formed by these other metals.

The anodic film always includes a thin, hard, compact "barrier" layer close to the metal. In non-solvent electrolytes this is the only film present and has a thickness in Angstrom units of about 13 to 14 times the anodizing voltage. In solvent electrolytes (i.e. those in which alumina is slightly soluble) an outer, much thicker, layer forms (Fig. 1). Normally the maximum thickness is about 0.0015 in. but special techniques enable films up to about 0.010 in. to be formed.

## Material Characteristics Affecting Anodizing

When work is to be anodized, and particularly when the decorative effect is important, the result depends upon the grade and quality of aluminium used; anodizing tends to emphasize, rather than conceal, any irregularity on the metal surface. The material suppliers and the anodizer should be consulted at the earliest possible stage, or at least advised of the material tentatively selected so that he can comment. Tables I and II are given to assist preliminary selection of material.

While thousands of tons of suitable alloys from the normal range are anodized with complete success every year, including embossed and patterned sheets of many types (available as standard items from the metal suppliers) most companies supply some standard materials in "anodizing quality" or "polishing quality" as well as a special range of alloys for bright anodizing. These may have been subjected to special processing during fabrication, and may possibly have additional minor alloying constituents, to improve the grain structure and facilitate brightening and/or anodizing. Recent developments include clad alloys which provide a more uniform appearance to the finished product. The cladding may either be the same or different from the basis aluminium; the technique of cladding is used to facilitate heavy

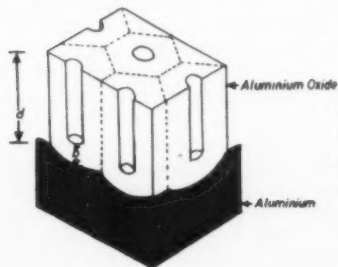


Fig. 1.

Pore structure of anodized surface showing layer of aluminium oxide. (not to scale).

\* Aluminium Development Association.

Table I

Anodizing Characteristics of Wrought Aluminium

Wrought Material Designation (B.S. 1470-1477)	Nominal Composition	Suitability for		
		Protective Anodizing	Anodizing and Dyeing	Bright Anodizing
I	99.99% Al	E	E	E
1A	99.8% Al	E	E	V - E
1B	99.5% Al	E	E	V
1C	99% Al	V	V	G
N3	Al-1½% Mn	G	G	M
N4	Al-2½% Mg	V	V	G - V
N5	Al-3½% Mg	V	V	G
N5/6	Al-4½% Mg	V	V	G
N6	Al-5% Mg	G	G	M
N7	Al-7% Mg	M	M	M
H9	Al-Mg-Si	V	V	G - V
H19	Al-Mg-Si types.	G	G	M
H20	More alloying elements than H9	G	G	M
H30		G	G	M
H11	Al-Cu-Mg-Si	G	G	M
H12	Al-Cu-Mg-Si-Ni	M	M(D)	U
H14	Al-Cu-Mg-Mn	M	M(D)	U
H15	Al-Cu-Mg-Si-Mn	M	M(D)	U
H18	Al-Cu-Mg-Ni	M	M	U
N21	Al-5% Si	G	G(D)	U

E = Excellent ; V = Very Good ; G = Good ;  
M = Moderate ; (D) = Only suitable for dark colours ;  
U = Unsuitable.

and uniform working of the metal forming the surface layer.

Where a good response is specially sought to chemical or electrolytic brightening followed by anodizing, special materials, not yet included in British Standards, may be used. In general, the brightness obtainable increases with increasing purity of aluminium, and it diminishes with increasing anodic film thickness, particularly on a metal of low purity. A similar effect is obtained with increasing purity of the aluminium used for the aluminium-magnesium (up to 2 per cent. magnesium)\* and aluminium-magnesium-silicon (¾ per cent. magnesium, ½ per cent. silicon optimum) alloys, which have better mechanical properties. When based on 99.99 per cent. aluminium, finishes brighter than chromium plate are obtained, but lower-purity alloys give brightness acceptable for much decorative trim and other applications.

\* Commercially available materials in this country based on aluminium of 99.99 per cent. purity include an aluminium-1½ per cent. magnesium alloy for general purposes, an aluminium-1½ per cent. magnesium alloy (of lower strength for use where ease of forming is the overriding consideration) and an aluminium - magnesium - silicon alloy comparable with HG. Similar materials are available which are based on 99.7 per cent. minimum purity aluminium.

The nature of the impurities is of major importance ; iron and titanium are most harmful.

Wrought aluminium-silicon alloys (notably those containing 4 to 6 per cent. silicon) have been developed because of the attractive grey finish which they acquire in the anodizing bath. The colour and intensity are dependent upon the thermal treatments of the material as well as upon composition and film thickness. They may be used as a cladding, either on metal of the same composition or on commercial purity aluminium or aluminium-1½ per cent. manganese alloy. Appropriate clad sheets engraved below the depth of the cladding and then anodized give in one operation grey and white patterns.

The addition to aluminium of other metals, such as manganese or chromium, whose oxides are distinctly coloured, gives further possibilities for the colouration of the oxide film (see B.P. 820,583).

### Modifications in Anodizing Solutions

In this country, sulphuric acid and chromic acid processes are by far the most important, while in a special category are the electrolytes giving "barrier-layer films." Oxalic acid anodizing processes, including the old "Ematal" (giving one type of enamel-like coatings) process, are used to a limited extent on the continent of Europe. These films have many properties similar to those of sulphuric acid films, but the process cost is higher. The undyed oxalic acid film, however, can be operated so as to give light shades of gold or bronze and analogous effects are obtainable with sulphosalicylic acid electrolytes (B.P. 850,576). In the space available here only a few of the most interesting modifications can be described. The most significant point is that the sulphuric acid type of anodizing solution remains the more versatile for general use and is supplemented by chromic acid (e.g. for crack detection and special decorative effects) and the barrier layer electrolytes (e.g. for electrolytic capacitors).

(a) *Chromic acid and sulphuric acid.* The traditional 3 per cent. chromic acid process gives a maximum film thickness of about 8 microns on aluminium with small amounts of alloying materials but only about 1.5 microns with the alloys containing several per cent. copper such as H15. Increase of chromic acid content of the bath up to 400 gm. per l. gives up to about 14 microns film thickness on pure aluminium. In practice use of 10 per cent. chromic acid at 130°F. and 30 volts enables films of about 8 microns to be reproducibly produced on a moderate range of alloys. A recent development with all these chromic acid processes is the use of cation exchange regeneration to oxidize the ineffective trivalent chromium to the hexavalent state. This is both practicable and economic for many installations and also keeps the

aluminium and other metallic impurities at a low level.

Addition of sulphuric acid enables much thicker films to be produced *e.g.* 50 gm. per l. chromic acid + 2.5 gm. per l. sulphuric acid at 110°F. and 30 volts gives a current density of 12 to 15 amps per sq. ft. and a film of over 20 microns in an hour\*. This film, however, contains 7½ per cent. sulphate and has only slightly better corrosion resistance than a film of the same thickness produced in sulphuric acid. The film is also considerably softer than a typical sulphuric acid film (15 per cent. acid, 15°C., 15 amp. per sq. ft., 1 hour) while a thin chalky surface film forms which is difficult to remove—this latter being a general characteristic of solutions using chromic acid above 100°F.

(b) *Sulphuric acid and Oxalic Acid.* Investigation of a range of compositions showed that the presence of oxalic acid enabled more abrasion-resistant films to be formed than in simple sulphuric acid solutions, or alternatively that the temperature of the mixed electrolyte could be higher than that of sulphuric acid without loss of abrasion resistance, hence easing tank cooling problems.

The mixed bath containing 60 gm. per l. sulphuric acid and 40 gm. per l. oxalic acid is of greatest value. Operated at 30°C. it gives films with abrasion resistance as good as those formed in 15 per cent. sulphuric acid at 21°C.\* The process is expensive, about 0.2 gm. of oxalic acid being used for each ampere-hour of current passed. In practice it is usually advisable to start baths at about 50 gm. per l. oxalic acid and to make calculated periodic additions. The bath has lower throwing power than the standard sulphuric acid bath which may be of importance with complex articles.

It is thought that no oxalic acid is retained in the film and that it therefore responds to sealing the same way and has very similar properties to the films formed in sulphuric acid.

(c) *Electrolytes for Thick Abrasion Resistant Films.* In addition to their use in industry for applications involving wear thick, hard anodic films are being tentatively developed for architectural and furniture applications, mainly in the U.S.A. The grey or brownish shades of the hard anodic coating are used as a design feature. Low concentrations of sulphuric acid (10 to 15 per cent. by weight) are favoured and low operating temperatures (about 0°C.) together with high current densities (15 to 400 amp. per sq. ft.) give the desired film. To obtain these conditions finally requires 40 to 100 volts and a good refrigeration

**Table II**  
Anodizing Characteristics of Cast Aluminium.

Cast Material Designation (B.S.1490)	Nominal composition	Suitability for		
		Protective Anodizing	Anodizing and Dyeing	Bright Anodizing
LM1-M	Al-7% Cu-3% Si-3% Zn	M	U	U
LM2-M	Al-10% Si-1½% Cu	M	U	U
LM3-M	Al-12% Zn-3% Cu	M	U	U
LM4-M	Al-5% Si-3% Cu	G	M	U
LM5-M	Al-5% Mg	V	V	G
LM6-M	Al-12% Si	M	U	U
LM7-M	Al-2% Cu-2½% Si-1% Ni	G	M	U
LM8-M, P, W, WP	Al-4% Si-½% Mg	V	G	U
LM9-W, WP	Al-12% Si-½% Mg	M	U	U
LM10-W	Al-10% Mg	G	M	M
LM11-W, WP	Al-4½% Cu	G	G	M
LM12-WP	Al-10% Cu-½% Mg	M	M(D)	U
LM13-WP	Al-12% Si-1% Cu-1% Mg-2½% Ni	U	U	U
LM14-WP	Al-4% Cu-1½% Mg-2% Ni	M	M(D)	U
LM15-WP	Al-2% Cu-1% Mg-1½% Si-1% Ni	G	G	M
LM16-W, WP	Al-5% Si-1½% Cu-½% Mg	G	M(D)	U
LM18-M	Al-5% Si	V	G(D)	U
LM20-M	Al-12% Si	M	U	U
LM21-M	Al-5% Si-3% Cu-2% Zn	G	M(D)	U
LM22-W	Al-5% Si-3% Cu	G	M	U
LM23-P	Al-2% Si-1% Cu-1% Ni	V	G	M
LM24-M	Al-8½% Si-3½% Cu	M	M(D)	U

V = Very Good ; G = Good ; M = Moderate ;  
(D) = Only suitable for dark colours ; U = Unsuitable.

system. The presence of oxalic acid is beneficial as indicated above.

Many operating processes are patented and considerable commercial usage has occurred in the last decade. The "Hardas" process†, now licensed in N. America as well as in Great Britain, uses a.c. superimposed on d.c., the proportions varying with the alloy being treated, the thickness of sheet and the speed of agitation. Some alloys, notably those high in copper, require an initial a.c. "strike." The "Hiduran" process‡ uses d.c. at constant wattage, to give a constant rate of heat extraction throughout the process,

\* A. W. Brace and R. Pulfreman. Lecture to Inst. Metal Finishing 19th October, 1959.

† Hard Aluminium Surfaces Ltd., Glasgow. Attention is drawn to British Patent 716,554.

‡ High Duty Alloys Ltd. Attention is drawn to British Patent 727,749.

minimizing the risk of "burning" at edges and corners. With this process it is necessary to use 30 per cent. acid for high copper alloys, such as H15. More recently in the U.S.A., the Toro process has been announced which is claimed to give a film 50 microns thick in only 2 minutes. The extremely high current density involved limits this process to suitable shapes and volumes of metal in which heat can be quickly dissipated. No further details of the process have yet been made available.

Thick, hard films may also be produced in other electrolytes at room temperature. Typical solutions are (a) 80 gm. per l. oxalic acid + 55 gm. per l. formic acid (b) 250 gm. per l. sodium bisulphate monohydrate + 100 gm. per l. citric acid. They are each operated at about 55 amp per sq. ft. and give films 100 microns thick in about an hour. Films up to at least 300 microns can be produced but the usefulness of the thicker films is limited by their mechanical fragility.

(d) *Special Solvent Electrolytes.* Other anodizing electrolytes include sulphamic acid, which is inferior to sulphuric acid in almost all respects; malonic acid, which compares in cost with d.c. oxalic acid anodizing, and gives hard films, coloured ochre or brown; phosphoric acid, suitable as a base for electroplating and sulphosalicylic acid, which has a number of disadvantages including difficult anodizing techniques.

Nevertheless this last acid has been proposed by the Kaiser Aluminium and Chemical Corporation for use in a mixed electrolyte (7 to 15 per cent. sulphosalicylic acid with 0.3 to 4 per cent. sulphuric acid + metal sulphates, e.g. ferric) which when used with a range of aluminium alloys gives an integrated series of light-fast colour anodized surfaces.

Techniques involving successive anodizing in a solvent-type electrolyte and then in a barrier-layer electrolyte have been proposed to improve corrosion-resistance. The term "double anodizing" is however more commonly applied in the nameplate and label industry to cover processes involving selective stripping and re-anodizing.

(e) *Electrolytes for "Barrier-Layer" Films.* For electrolytic capacitors and for anodizing vacuum-deposited aluminium, barrier-layer electrolytes, such as boric acid, ammonium tartrate, ammonium borate or ammonium dihydrogen phosphate, are used in which alumina is insoluble.

The films formed in barrier-layer electrolytes are much thinner than those formed in electrolytes which have some solvent action on the film as it forms. The use of distilled water is more important with non-solvent anodizing solutions than with solvent type solutions. The film thickness depends almost entirely on the forming voltage

being approximately 13 to 14°A per volt (10,000°A = 1 micron). The choice of voltage depends on other factors also, such as the bath resistance and the need to avoid sparking. Adequate films are usually formed in about 45 min.: the need for forming periods greater than twice this usually indicates defects or dirt in the metal surface and the product is unlikely to make a satisfactory electrolytic capacitor. Unlike the films formed in solvent electrolytes, subsequent sealing is not needed with barrier-layer films; they are merely washed and dried.

With boric acid, solutions from 4 per cent. boric acid up to saturation are used, to which 0.05 to 0.5 per cent. borax is added. Avoidance of chloride impurity is important: as little as 0.01 p.p.m. may prevent oxide formation. Temperatures range from 70° to 100° C. (158°F. to 212°F.). Current density is typically 5 to 10 amp per sq. ft. initially and this requires high voltages, from 50V (with the most concentrated solution) to 750V; the current then falls to a low level. The film, which is free from borates, has a thickness directly proportional to the final forming voltage.

*Ammonium Tartrate* solutions normally contain 3 per cent. tartaric acid with ammonium hydroxide added to bring the pH to 5.0 to 5.5. The current density is initially about 20 amp. per sq. ft. but decreases in about a minute to a few milli-amp per sq. ft. after which the film virtually ceases to grow.

### Development of Brightening Processes

In this category are included both chemical and electrochemical processes either of which types may involve removal of small or relatively large quantities of metal (up to several thousandths of an inch). Mention has already been made of special materials developed for their brightening characteristics. In addition a useful lustrous finish can be obtained on many of the General Engineering Series of Aluminium Alloys (B.S. 1470-77, 1490). The suitability of these alloys for brightening with subsequent anodizing is given in Tables I and II.

Selection of polishing processes can not readily be tabulated. Phosphoric nitric base chemical smoothing solutions (Phosbrite 159: Alupol V) give good results on a wide variety of materials but carbonate/phosphate electrobrightening solutions (Brytal) or nitric/bifluoride chemical smoothing solutions (Erftwerk) are preferred for super-purity aluminium and its alloys. Other solutions, notably the phosphoric acid type (e.g. Alkubrite) or sulphuric/chromic acid type (e.g. Aluflex) electro-smoothing solutions and dilute nitric/fluoride chemical smoothing solutions (e.g. "S") have specific advantages but they have less commercial

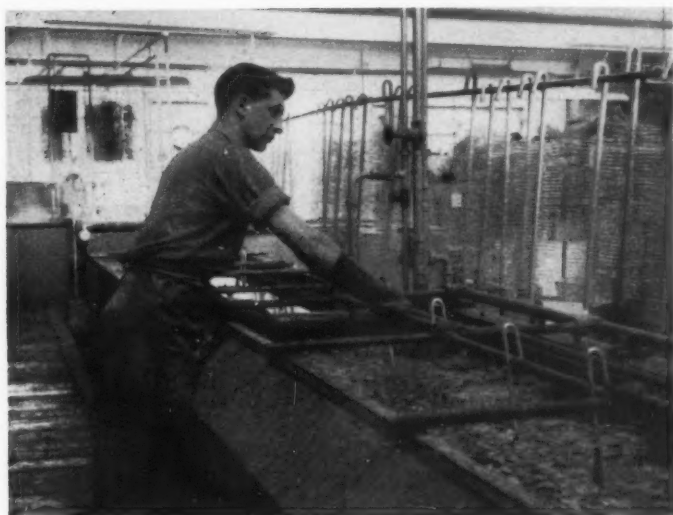


Fig. 2 Typical jobbing plant with tanks arranged end on, showing need for close access.

(Courtesy Acorn Anodising Co., Ltd.)

use at present in this country. Space however does not allow full treatment of this subject here.

The technique of the subsequent sulphuric acid anodizing process is also important. Recent work by Cochran *et al* (*Modern Metals* 1960, Oct.) indicates that significantly higher reflectivity is retained in a 15 per cent. sulphuric acid bath using 12 amp per sq. ft. current density if the temperature is 84 to 88°F. (requiring 10 to 12 volts) than if it is 70°F. (requiring 15 to 16 volts). This higher temperature film will however be slightly less abrasion resistant.

### Plant Development

It is intended here to limit discussion to the overall considerations arising from the expansion of the hand jobbing anodizing industry to the semi-automatic, automatic and continuous processes in use today.

With jobbing plants the main requirement is ease of access to the tanks and these are therefore arranged separately and end-on or in small groups as shown in Fig. 2. For semi-automatic work and for large components where automatic hoists are available the side by side arrangement shown in Fig. 3 is preferable.

Baths are available for anodizing sheets up to about 26 ft. x 9 ft. and extruded lengths up to about 32 ft. Dimensions near these maxima often give rise to difficulties in ancillary processing baths, and it is recommended that the size of sheets should be limited to 12 ft. x 8 ft. where possible.

The use of anodizing in mass-production industries, such as those concerned with the pro-

duction of washing machines, refrigerators, nameplates, motor-car trim, etc. has made it inevitable that attention should be given to producing anodic finishes in automatic plants, and a number of plants are in existence in various countries.

One of the main advantages of an automatic plant is that owing to the elimination of the human element in operations involving a series of treatments in various baths, the final product should theoretically be more uniform and of better quality. However, the quantity of work which may be processed in an automatic plant is so great that some arrangement is necessary for continuous replenishment of solutions. While close control of temperature may be possible, uneven draining and unequal cooling during transfer from treatment to rinse tank may affect the final appearance. Carry-over of solution can rapidly cause contamination unless rinsing is adequate; a spray rinse is preferred. When brightening solutions are introduced into the automatic line it is necessary to ensure rapid rinsing, tolerance times between removal from the bath and effective rinsing are for many solutions only 30 seconds or a minute.

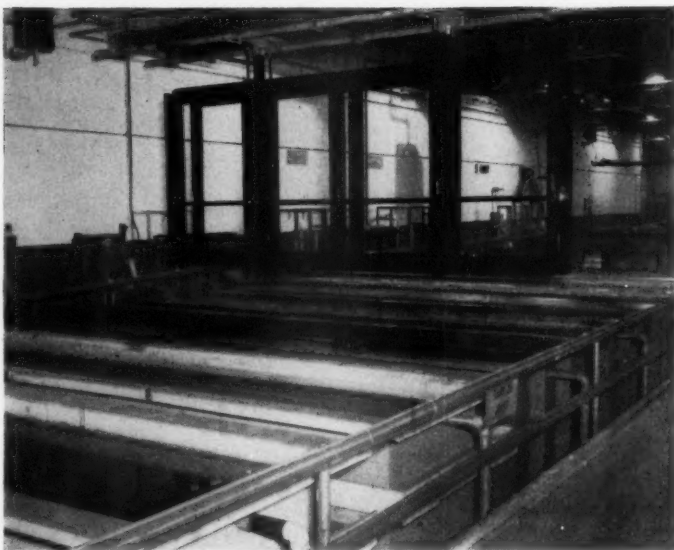
One small innovation of particular importance in automatic plants is the use of titanium in jigs, eliminating the need for removal of anodic oxide after each treatment cycle.

### Continuous Anodizing

Where aluminium is available in continuous lengths, for example, coils of wire or strip, it can be driven or pulled over a series of pulleys or rollers so that it passes through each treatment bath or rinse. The aluminium moves at a uniform

Fig. 3.—Automatic hoist in operation, conveying large component between tanks arranged side by side. This installation is for a colour anodising operation for large building panels.

(Courtesy Reynolds Metals Co., New York).



speed. Speed is kept constant for any particular film thickness, but the speed is adjustable, of course, to provide for different film thicknesses. The time of dwell in each bath is predetermined by the effective bath lengths. The solutions and operating conditions are basically as for ordinary sulphuric acid anodizing.

Continuous processes are economically applied to the anodizing of long lengths of wire, strip and foil to produce a product suitable for applications where electrical insulation or the ability to take dyes is coupled with the need for some corrosion resistance and a ductile coating. The product is used for electrical conductors and for mass-production lines where forming takes place after anodizing, as in the manufacture of radio, trim cans, and bottle tops. The maximum anodic film thickness suitable for forming varies according to the processing conditions, but is usually about 4 to 6 microns. The anodic film is often much thinner; for example, canning strip has a film 0.2 to 0.4 microns thick as a base for lacquer.

No mechanical pre-treatment is normally applied to wire and decorative strip. The continuous anodizing of foil and thin strip is mainly used for electrical insulation between turns of windings. Low voltage-breakdown characteristics at the edges have been overcome in several ways, e.g. by a combination of mechanical and chemical polishing prior to anodizing. Chemical treatment may include cleaning or etching, the solution concentrations being high to keep the time of treatment low. Inclusion of brightening processes gives some difficulties in practice. In one process for

strip to be very thinly anodized, the addition of methyl cellulose to the anodizing bath obviates prior chemical cleaning.

For wire, anodizing speeds generally in excess of 35 ft. per min. are necessary for economic reasons. To keep baths within reasonable size, this means that anodizing periods must not exceed about 45 sec. and to form a film of 5 micron thickness will require a current much higher than the wire could carry in air. Several processes overcome this by so-called liquid electrical contacts in which the current is induced in the wire after it has entered the electrolyte in one bath prior to passing into the main tank. Current densities are usually 300 to 900 amp per sq. ft. Both d.c. and a.c. processes are operated but one commercial plant uses a combination of the two. When d.c. is used the wire may be made cathodic while in the entry chamber, and anodic as soon as it passes through a small nozzle into the main tank. With a.c., two electrode chambers may be used each being connected to one end of the secondary winding of the transformer; the electrodes consist of one or more pairs of wires equally distributed between the two chambers.

For strip, slower speeds of 15 to 35 ft. per min. are used. While liquid contacts are used, often ordinary contact rollers together with a mechanical shield at the point of entry into the solution are entirely satisfactory; if the voltage exceeds about 25 volts, however, sparking may occur where the strip enter the liquid and cause pitting.

(Continued in page 151)

### Some Recent Advances in Anodizing Practice and knowledge

(Continued from page 150)

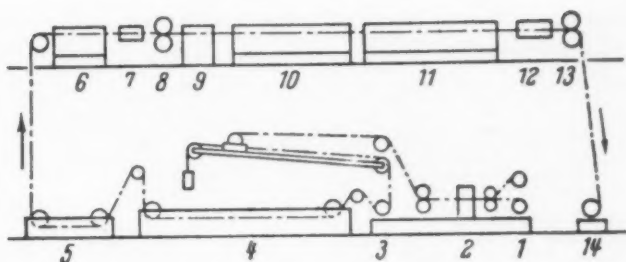


Fig. 4. Layout of plant for continuous anodized strip using the lacquer sealing process. This is the plant installed at Nordisk Aluminiumindustri. (1) Uncoiling. (2) Joining. (3) Power supply. (4) Anodizing. (5) Rinsing. (6) Drying. (7) Cooling. (8) Tension rolls. (9) Lacquering. (10) Drying. (11) Stoving. (12) Cooling. (13) Tension rolls. (14) Coiling.

A high sulphuric acid concentration is desirable both for maximum conductivity (attained at 30 per cent. acid) and to give a flexible coating. The use of a.c. gives more flexible coatings than d.c. Various additions to the bath have been proposed but the most popular, chlorides, are inherently undesirable as they may be trapped in the coating and so reduce the corrosion resistance. These baths need cooling by suitable means and are usually worked slightly above room temperature.

Wire from 0.0008 in. up to about  $\frac{1}{4}$  in. diameter can be processed. Strips of most gauges and widths can be processed. Separate plants are needed for materials of markedly different dimensions. Material processed must not be too soft or it will stretch during processing, or too hard or it will be difficult to coil. For many purposes  $1C-\frac{1}{2}H$  is suitable.

For strip, treatment after anodizing may follow the normal sequence, including dyeing if required, and sealing with process modifications to suit the speed of the process. Thus a significant degree of sealing in boiling water may be obtained

in as brief a period as 30 sec but longer or additional treatment is preferable. Nickel acetate sealing is often preferred as it is claimed to be less sensitive to impurities (notably phosphate, fluoride and silicate) and also to be more speedy in action. The speeds used for wire anodizing usually preclude such treatment but the wound coils are sealed by immersion for 3 to 4 hours in boiling water, followed by drying at 82°C. (180°F.) for a similar period.

Continuous anodized strip is however specially suited to the "lacquer sealing" patented Norwegian process (Fig. 4). After anodizing in the bath containing methyl cellulose the strip is washed, dried and roller coated with a special synthetic lacquer which can be polymerized in a very short time at an elevated temperature.

### PROGRESS OF MOND LABELLING SCHEME

A REPORT has recently been issued by the International Nickel Co. (Mond) Ltd. regarding the activities of their labelling scheme for chromium plate for the first half of 1961.

By direct mail two leaflets are being sent to 13,000 manufacturers. The scheme will also be given extensive advertising in the press and will be publicized at various exhibitions and at the Llandudno conference of the Institute of Metal Finishing. In addition, the scheme will be featured in many television advertising programmes in London, the Midlands and the North. Later plans will include participation in the Motor Show and further television advertising.

The number of signatories to the scheme is now over 150.

### ANODIZED ALUMINIUM ON T.V. CENTRE

WORK is nearing completion on the 10-floor administrative block of the Granada TV centre, Manchester. Built at a cost of £500,000, the new building comprises phase IV of the development plan of the centre. The new building, which is surmounted by a residential penthouse, and is built on the beamless concrete floor principle, has front and rear walls of curtain wall construction. By this method a prefabricated anodized framework to take the windows and decorative panels is hung from floor to floor and backed by a concrete wall. The curtain walling was supplied by Templewood Hawkesley. Unilock Partitions Ltd. supplied the partitions for the block. Unilock is a patented, light-alloy partition, finished in a coloured grained plastic and Georgian wired glass.

## Multilayer Metal-Ceramic Coatings For High-Temperature Protection

(Continued from page 144)

### Surface Preparation for Application

Surface preparation capable of providing a suitable base for the initial coating material is mandatory for success of the application, and resultant use. The graphite must be suitably roughened and cleaned before excellent wetability can be attained with the metallized molten metal or flame-sprayed plastic oxide particles.

Light roughening was accomplished in each case by use of wire brushing as previously described. Blasting operations not only tended to contaminate the base material, but metallographic examination disclosed the formation of numerous surface and just sub-surface cracks as a result. These cracks in some cases, caused premature failure of the coated part.

After roughening, the parts are air-blast cleaned with clean dry air applied across the surface of the part. Loose graphite hinders reliable adherence, as shown by the use of colloidal type graphites as a mask for use in metallizing processes.

Once suitably roughened, the part is next diffusion treated (siliconized) and coated, or as in the case of the rocket nozzles coated. Exceptional care is taken at all times to avoid handling with bare hands or the use of any oily substance in contact with the graphite to be coated. With a graphite base, as with a metal base, cleanliness is necessary for good adhesion.

The ability of multilayer metal-ceramic coatings to withstand severe erosion, thermal shock, and high temperatures in a reducing atmosphere has been proven by its use in protecting rocket blast deflectors, afterburner rakes, rocket nozzles, and the internal walls of a sectional burning solid propellant rocket motor. As evidenced, preliminary post diffusion treatments of the metal-ceramic coated part are providing aid in improving its oxidation resistance at high temperatures.

By use of other application equipment such as the plasma gun, vapour deposition chamber, vacuum impregnation chamber and hydrogen reduction furnace, as well as other or additional processes such as impregnation plus diffusion, thermal decomposition, hydrogen reduction, and the electrolytic processes of electrophoresis and electroplating, a new type of tailored multilayer metal-ceramic coatings can be developed. Possibly one or more of these coatings will provide very extended oxidation resistance.

Metal-ceramic laminated coatings of the type described, although relatively new, have proved capable of extending the life of the base materials

under severe environmental conditions. Considerable additional test work must be performed before the optimum coating system for any service condition can be selected with assurance. Until the completion of such tests only those coating systems which are found to be adequate to pass the environmental requirements will be utilized.

### SHARING SCIENTIFIC EQUIPMENT

**A** SCHEME for sharing scientific equipment between industrial companies in South-East England has recently been announced by the FBI London and South Eastern Region. It is believed to be the first arrangement of its kind in England and Wales if not in the United Kingdom.

A large proportion of the 126 industrial firms represented in the Region's Research Directors' Discussion Group have submitted two lists of items of equipment—one of items they are prepared to make available to other companies; the other of equipment to which they would like to have access. These lists are now being circulated to all members of the Group with the suggestion that "offerers" and "seekers" should get in direct contact. It is hoped that other companies will be moved to participate as a result of this initial circularisation. The idea arose from a suggestion made by a member of the Group.

### INSTRUMENTS AND CONTROLS EXHIBITION

**T**HE 1961 Midlands Exhibition of Instruments and Controls for Industry, Education and Research will be held at the Y.M.C.A., Snow Hill, Birmingham, from April 25 to 28 next. This annual exhibition is the only one of its type to be held in the Midlands. Further details may be obtained from A. M. Lock and Co. Ltd., Prudential Buildings, 79, Union Street, Oldham, Lancs. Telephone: Oldham Main 0333.

### DANISH ELECTROPLATERS VISIT GREAT BRITAIN

**T**WENTY-SIX members of the Danish Union of Electroplaters will arrive on April 16 for a seven-day visit to Great Britain, as guests of W. Canning and Co. Ltd., Birmingham.

Arrangements have been made for the members to inspect modern automatic and semi-automatic electroplating equipment at Midland firms including Midland Electric Manufacturing Co. Ltd., Harrisons (B'ham) Ltd., Wilmot Breeden Ltd., and Laystall Cromard Ltd. The Danish visitors will also see the laboratories and engineering works of W. Canning and Co. Ltd. before they depart on a tour of places of interest including Stratford-on-Avon, Oxford, Windsor, Hampton Court, etc.

## FINISHING

## NEWS REVIEW

## INSTITUTE OF METAL FINISHING

## Details of Annual Conference Programme

AS announced in a previous issue of this Journal, the twelfth Annual Conference of the Institute of Metal Finishing will be held at Llandudno from May 2 to 6, 1961, with the technical sessions taking place in the Grand Hotel. In addition to the presentation and discussion of technical papers there will be a number of planned social functions, including a Conference dinner and dance, a visit to a works, and a special programme for the ladies. Full details of the programme are given below:

**Tuesday, May 2. Grand Hotel**  
Evening—Coaches will meet trains arriving at Llandudno between 3.30 and 5.30 p.m. Registration at Conference Office.

**Wednesday, May 3. Grand Hotel**  
9.30 a.m.—Official Opening of Conference.

followed by 1st Technical Session—Nickel and Chromium.

- 1 "The Influence of Addition Agents on the Composition of Nickel Deposits." By A. H. DuRose.
- 2 "Levelling Action in Electrodeposits of Nickel and Acid Copper." By S. A. Watson.
- 3 "The Corrosion of Decorative Nickel Chromium Coatings: Metallographic and Potential Study." By G. N. Flint and S. H. Melbourne.

Afternoon

2nd Technical Session—Process Studies.

- 4 "Electrodeposition of Ruthenium." By F. H. Reid and J. C. Blake.
- 5 "Electrodeposition in Manufacture of Plain Bearings." By P. G. Forrester.
- 6 "Experience with an Ion-Exchange Process for the Recovery of Water Polluted in Metal-Finishing Operations." By A. A. L. Pearson and G. G. Parker.

## Café Royal

Evening—Reception and Dance by invitation of the Chairman and Members of Llandudno Urban District Council.

**Thursday, May 4. Grand Hotel**  
Morning—3rd Technical Session—General Research.

- 7 "An Improved Technique for Measuring the Cathode Overpotential During Electrodeposition." By J. K. Prall and L. L. Shreir.
- 8 "Influence of the Composition and Heat Treatment of Carbon and Low Alloy Steels upon Response to the A.R.D.E. Smoothing Process." By W. A. Marshall, V. J. Sanders and R. A. F. Hammond.
- 9 "Influence of Preplating Processes on the Porosity of Electrodeposits on Steel." By M. Clarke and S. C. Britton.

Afternoon—Visit to the Works of A.E.I. (Hotpoint) Ltd.

## Grand Hotel

Evening—Seventh Hothersall Memorial Lecture by A. Brenner. Dr. Abner Brenner is chief of the Electrodeposition Section of the U.S. National Bureau of Standards.

**Friday, May 5. Grand Hotel**  
Morning—4th Technical Session—Selection and Testing of Coatings.

- 10 "A Calorimetric Method of Coating Thickness Measurement." By G. Krijl and J. L. Melse.
- 11 "Atmospheric Exposure Tests on Anodized Aluminium." By J. A. Whittaker and J. M. Kape.

- 12 "Selection of Finishes for Communications Equipment." By W. Marchand.

Afternoon—5th Technical Session—Selection and Testing of Coatings—continued.

- 13 "New Developments in Equipment for Painting." By A. Newcomb.
- 14 "Selection of Finishes for Protection and Decoration in the Motor Industry." By R. J. Brown.
- 15 "The Use and Value of Laboratory Tests of Paint Coatings." By J. A. W. van Laar.
- 16 "The Selection and Testing of Finishes for Domestic Appliances." By R. Warburton.

## Winter Gardens

Evening—Reception by the President and Mrs. Harvey followed by Conference Dinner and Dance (Evening dress or dinner jacket preferred).

**Saturday, May 6.**

CONFERENCE CLOSES.

## LADIES' PROGRAMME

**Wednesday, May 3.**

Afternoon—A coach trip through the Welsh Mountains to Llanberis and Snowdon via Bettwys-y-Coed and Lake Peris. Tea at Llanberis at the foot of Snowdon.

**Thursday, May 4.**

Afternoon—A coach tour to Beaumaris and the Isle of Anglesey crossing both the Conway and the Menai bridges through Penmaenmawr and Llanfairfechan into Bangor; tea at Beaumaris.

**Friday, May 5**

Afternoon—A Surprise Item.

All communications regarding the Conference should be addressed to the Conference Secretary, Institute of Metal Finishing, 32 Great Ormond Street, London, W.C.1.

## £250,000 PLATING PLANT FOR NUFFIELD

ONE white-coated worker, with a flick of an index finger, will be able to control the Nuffield Organization's new £250,000 Deinert plating plant.

This fully-automatic plant, housed in a vast red-and-white tiled hall at Morris Motors' Radiators Branch in North Oxford, began producing the chromium trim for all Nuffield cars during March.

It is the most advanced of its kind in Great Britain and is probably unique in that it deposits copper, nickel and chrome in an unbroken series of operations. Once parts to be plated have been fed to the machine, it is not normally necessary to remove or touch them until the plating cycle is completed some two hours and ten minutes later. This reduction in the amount of handling normally necessary means fewer damaged—and thus rejected—components and minimum wastage of plating materials.

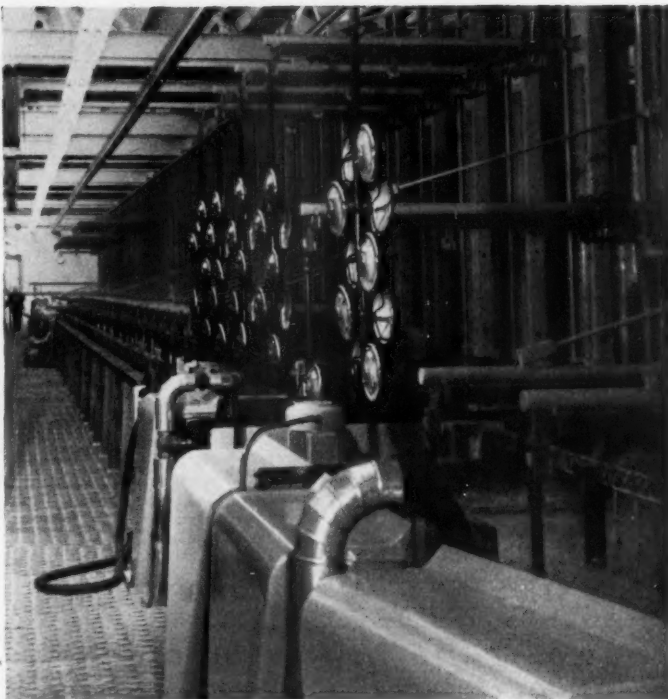
Basically, the plant consists of 36 welded steel vats, the largest of which can hold 12,000 gal., arranged in an 177 ft. "U" shape. Overall width is 20 ft., height 19 ft. 10½ in. and weight with solutions about 350 tons.

The plant uses about 160,000 gal. of pure water per day and sufficient electricity to supply all the needs of a village of about 750 houses. It will deliver 1,000 sq. ft. of high quality chromium plate an hour (about 1,000 Morris Minor hub caps, for example). This entails depositing 60 lb. of copper and 60 lb. of nickel each hour—about 10 tons of each during a working month. All plating work is carried out to strict British Motor Corporation standards.

Although the amount of water may seem large, it actually represents a considerable economy of the normal requirements for such a plant: automatic pressure sprays, to prevent components drying while being transferred from one vat to another, are extensively used to conserve water.

The machine can handle 100 jigs an hour, each carrying a maximum weight of 55 lb. The jigs, two per flight bar, move forward every 72 seconds.

Of the 36 vats, 32 are filled with degreasing solutions, water and acids for cleaning, swilling and washing. The remainder hold 2,750 gal. (alkaline copper), 9,200 gal. (acid copper), 12,000 gal. (nickel) and 4,500 gal. (chromium). Provision has been made for parts to be removed for polishing between the copper and nickel stages, if required.



*Looking down one side towards the loading point of the plating plant. Hub caps leaving the copper vats are moving towards the cleaning vats in the foreground prior to Ni Cr. plating.*

Total installed capacity of the plant, which uses more than 50 electric motors, is two megawatts. Current is drawn from the Southern Electricity Board's 11,000 volt lines, stepped down to 400 volts, rectified and then fed to the plating vats at 3,000 amps. (alkaline copper), 30,000 amps. (acid copper and nickel) and 24,000 amps. (chromium) at six to eight volts.

As far as practicable, ancillary equipment has been grouped close to the plant, partly for economy of operation and partly because space is at a premium at Radiators Branch. All major electrical equipment (transformers, transformer/rectifiers etc.), have been mounted on a gallery to one side while the ten storage tanks, which hold reserve solutions and liquids from the vats during routine inspections and maintenance, have been squeezed into one corner. The only equipment not adjacent to the plating shop are the factory's effluent plant and the boilers which supply

steam for the heat exchangers that maintain the solutions at constant temperatures.

Save for the loading and unloading, the plant is fully automatic, and, by virtue of its many built-in safeguards, is mechanically foolproof.

### South African Contract

**B**ROOKHIRST Igranic (a company in the Metal Industries Group) has received an order to supply control equipment for a new factory which has been engineered by W. J. Fraser of Romford and which is being erected by South African Titan Products in Umbogintwini. The factory is scheduled to produce titanium dioxide which forms a basic chemical in the manufacture of paint and paper. The raw material used in making titanium dioxide is ilmenite of which there are large deposits on the southern coast of Natal.

## ANALYSIS OF TIN AND ITS ALLOYS

**F**IVE methods in a new British Standard series of methods for the sampling and analysis of tin and tin alloys have just been published. Another seven methods will be issued in due course.

The complete series is being published under the collective number and title, B.S. 3338, "Methods for the sampling and analysis of tin and tin alloys," each part being issued as a separate booklet.

The parts published so far are: Part 1—Sampling of ingot tin; Part 2—Tin in ingot tin (aluminium reduction method); Part 7—Silver (volumetric: ammonium thiocyanate method); Part 11—Tin in solders (nickel coil reduction method) and Part 12—Sampling of solders.

The methods are intended for the sampling and analysis of the grades of metal covered by B.S. 3252, "Ingot tin," B.S. 218, "Soft solders" and B.S. 3332, "White metal bearing alloy ingots."

Part 1 specifies selection of ingots, preparation of surfaces, method of taking samples by means of sawing and precautionary measures against contamination of the sawings.

Part 2 lays down a method for determining tin in ingot tin having a tin content from 99.0 to 99.8 per

cent. Part 7 gives a method for determining silver in solders having a silver content of up to 3 per cent, and Part 11 contains a method for determining tin in solders having a tin content exceeding one per cent. Each of these three parts specifies reagents required, recommended methods of sampling and test procedure.

Part 12 specifies selection of ingots, bars, sticks or wire, preparation of sample, preparation of surfaces, methods of taking final samples by means of sawing or filing, and precautionary measures against contamination of the samples.

Copies of these standards may be obtained from the British Standards Institution, Sales Branch, 2 Park Street, London, W.1, Part 2 price 4s. and Parts 1, 7, 11 and 12, price 3s. each. (Postage will be charged extra to non-subscribers).

### Change of Telephone No.

**T**HE telephone number of Kabi (Electrical & Plastics) Ltd., has been changed to Potters Bar 53444. Callers from London should dial PR followed by the five figure number.

### ON-THE-ROLL DRUM RACK

**S**EVERAL problems were solved at once when the Ford Motor Company had this special on-the-roll drum racking designed to store nearly 7,000 gal. of paint in five-gallon drums at their Doncaster plant. Dexion slotted angle was used for the tailor-made installation, which now affords ready selectivity between brands and colours, first in—first out rotation of stock—and room to walk about.



## INTERPLAS 61

**A**LREADY over 350 firms from thirteen countries have arranged to display their products at this year's International Plastics Exhibition—INTERPLAS 61—in London from June 21 to July 1. Countries represented include Austria, Belgium, Canada, Denmark, Finland, France, Great Britain, Germany, Holland, Italy, Sweden, Switzerland, and the U.S.A.

Visitors will see assembled in the three halls of Olympia, machinery, materials and products together representing the complete picture of world plastics progress. Displays of fabricating and ancillary equipment demonstrated by the major machine builders will highlight the latest advances in plastics moulding, vacuum forming, extrusion and blow moulding. The array of modern plastics materials will include an extensive range of typical end uses, both current and potential, and many new materials now under development will be shown.

Tickets and further information can be obtained from Iliffe Exhibitions Ltd., Dorset House, Stamford Street, London, S.E.1.

### NEW METHOD FOR SPALLING RESISTANCE OF ENAMEL

**A**STM Committee C-22 on Porcelain Enamel has completed a method to determine the spalling resistance of porcelain enamel on aluminium. This method is based on immersion of coated panels in an ammonium chloride solution. It is claimed that there has been no authenticated case in which specimens have passed this test and then failed in service.

A method to determine the thermal stress and strain produced by porcelain enamel in metal substrates is under development. An enamelled strip of steel acts like a bimetallic thermostat, and a differential transformer is used to measure the specimen deflection. In this manner, the stress behaviour of various combinations of enamel coatings and base metals can be determined. This method may also be used to study the differential thermal expansion behaviour of dissimilar metals bonded together by ceramic adhesives.

Interlaboratory work is continuing on the method of determining the coefficient of expansion of porcelain enamel. Also being investigated are methods for impact, continuity of coating by high- and low-voltage breakdown tests, and the sag characteristics of steel during firing of porcelain enamel coating.

## TECHNICAL and INDUSTRIAL APPOINTMENTS

Mr. K. A. Robinson, A.M.Brit. I.R.E., has been appointed to the board of **Lancashire Dynamo Electronic Products**, (a company in the Metal Industries Group).

Before joining the company, Mr. Robinson was concerned with the development of industrial electronic equipment with the English Electric Company at Stafford.

\* \* \*

As part of the policy of **The Ruberoid Co., Ltd.**, of building up their development department, Dr. H. A. F. L. Kremer, B.Sc., Ph.D., has been appointed to fill the newly-created position of chief development chemist.

Mr. D. S. Symons, A.M.I.Mech.E., M.I.P.E., has joined the company as Chief maintenance engineer at their Brimsdown, Enfield, factory.

Another new appointment at Brimsdown is that of Mr. C. W. High as electrical engineer.

\* \* \*

E. C. Gill, president of The Canada Life Assurance Company, has been elected a member of the Advisory Committee of **The International Nickel Company of Canada, Ltd.**

A native of Kingston, Ontario, Mr. Gill graduated with a Bachelor of Arts degree from Queen's University in 1923 and received an Honorary Doctor of Laws degree there in 1957. He is Chairman of the Board of Trustees of that University. He is a Fellow of the Society of Actuaries, a member of the Board of the Life Insurance Association of America, and a past President of The Canadian Life Insurance Officers Association.

\* \* \*

**The Paint Manufacturers' Joint Executive Council** has appointed Mr. Keith S. Flory as director and he will commence his duties on May 1, 1961.

Mr. Flory has had considerable experience in the paint industry, having occupied senior executive appointments with two important companies, during which time he has rendered valuable service on industry committees, including the Building Paints Advisory Council.

**ESSA, Electroplating Engineers S.A.**, Geneva, Switzerland, announce that, owing to the considerable extension of their activities, they have appointed Mr. D. E. Weimer of England as liaison officer, to work alongside their distributors in Denmark, Great Britain, Holland, Norway, and Sweden. Mr. Weimer has been employed by M. L. Alkan Ltd., the ESSA distributor in the United Kingdom, for the last seven years as chief development chemist.

He is known as an expert in the metal finishing field, and is an active member of the Institute of Metal Finishing. He has had published numerous technical articles and has also given many papers to branches of the I.M.F.

Mr. Weimer is also very familiar with plating practices outside his own country, having travelled extensively in the United States and Europe.

\* \* \*

**CIBA (A.R.L.) Ltd.** announce the following changes in their sales organization: Mr. D. A. Leitch, formerly Scottish area manager, is appointed Southern area manager; Mr. L. W. Taylor becomes Scottish area manager; Mr. J. M. Turner becomes Midlands area manager. Mr. S. Hopwood continues as Northern area manager, and Mr. G. Grant as North Midlands area manager.

\* \* \*

It is announced by **Ferro Enamels Ltd.** of Wombourne, Wolverhampton, that Mr. C. Vickers, A.M.I.E.E., A.M.I.Mech.E., has been appointed general sales manager.

His introduction to the enamelling industry came with his appointment as chief executive officer of a prominent manufacturer of holloware and associated products in the Midlands. Since that time he has taken a keen interest in the promotion of vitreous enamel.

In his capacities of director of the Vitreous Enamel Development Council, and Vice-Chairman of the Hardware Manufacturers' Association, he has been prominently concerned in publicizing enamelled products.

In recent times he has pioneered the manufacture and promotion of enamelled aluminium in this country, having put down and operated the first purpose-built plant.

\* \* \*

Mr. A. L. Howard, has joined **Blundell, Spence and Co. Ltd.** (Industrial Division) as transport finishes representative in an area covering Shropshire, Staffordshire, Warwickshire, Worcestershire and Herefordshire. He will be attached to the Blundell depot at West Bromwich.

Mr. Bernard J. Nicholson, previously joint vice chairman with Mrs. Vera Lilley, has been appointed the first president of the **Berger, Jenson and Nicholson** group of companies and remains a member of the board. Mr. R. Ashley Hall, a director of the group, becomes joint vice-chairman. Mr. Stanley G. Barnett, director, has been appointed deputy to the joint managing directors Mr. J. Nicholson and Mr. W. J. Vines.

\* \* \*

It is announced that Mr. Denys S. Asbury (Hanover House) has been appointed assistant secretary to the **Laporte Group** of Companies.

## OBITUARY

THE death occurred recently of Mr. Benjamin L. Watson, joint managing director and vice-chairman of **Teleflex Products Ltd.** of Basildon. Mr. Watson was a Teleflex executive for twenty-four years. He joined the company in 1937 as an accountant and in 1943 was appointed secretary. In 1947 he became a director and in 1952 was made joint managing director.

Mr. Watson was one of those responsible for the expansion of Teleflex. When he joined the firm their chief business was in the design and manufacture of controls for aircraft. Now Teleflex conveyors, remote control window gear, actuator and marine steering controls constitute an important part of the business.

Mr. Watson was also chairman of the Basildon Industrial National Savings Group.

## Scottish Branch for Institution of Chemical Engineers

THE President of the Institution, Mr. W. K. Hutchison, C.B.E., recently presided at the Inauguration of the Scottish Branch of the Institution in Edinburgh, when the following Branch Committee was elected: Chairman, Mr. T. Flavel; Vice Chairman, Prof. A. W. Scott; Hon. Secretary, Dr. D. M. Wilson; Hon. Treasurer, Dr. R. G. Gardner; Committee, Mr. L. H. Cleat, Mr. R. Hendry, Mr. V. E. Mahaffy, Mr. C. S. McBain, Mr. D. W. Rowbotham.

There has been in Scotland for a number of years an informal Group, but in view of the success of this Group and the increase in membership in Scotland, the Council approved a petition from Corporate Members in Scotland requesting Branch status.

## NEW ELECTROLUMINESCENT DISCOVERIES

NEW discoveries have brought the industrial and equipment lighting of the future—electroluminescence—one step nearer reality.

This lighting illuminates whole metal sheets by the use of phosphorus, which lights up when an electric current is passed through it. No filaments or elements are needed.

Dr. Peter W. Ranby (chief chemist at the Lighting Laboratories of Thorn Electrical Industries Ltd.) and his research group, have developed a method that increases the brilliance of the plates more than three times.

Some of the new plates were exhibited at the Tenth Electrical Engineers Exhibition at Earls Court recently, on the stand of a Thorn subsidiary, Atlas Lighting Ltd.

As far as it is known, this is the only British company developing the "ceramic on metal" type of electroluminescent product with a lamp

built upon a steel sheet which forms the back electrode. The phosphorus is embodied in a vitreous enamel, fired at high temperatures. The lamp is completed with a conducting transparent layer of tin oxide to form the second electrode, and a protective clear overglaze.

It is claimed that, for ceramic lamps, they are more robust and versatile than the "organic on glass" lamps. The latter consists of a glass sheet on which a transparent conducting film of tin oxide is deposited, followed by the phosphorescent layer in an organic resin binder. The capacitor is completed by a second electrode of evaporated metal and the assembly sealed with paraffin wax to exclude moisture.

These lamps are suitable for aircraft instruments, car instruments panels and radio tuning dials; luminous clock faces and map readers are already in production.

## CONTINUOUS COATING MACHINES

HENRY Simon Ltd. announce the conclusion of a licence agreement with the Midland-Ross Corporation of Cleveland, U.S.A., for the manufacture and sale of Waldron web processing machinery. A range of machines will be marketed in Western Europe and the Commonwealth under the name of Simon-Waldron.

The main result of this agreement is the manufacture in Europe of a well-established line of web processing machinery used for the application of finishes and various kinds of treatment to any materials which can be uncoiled in a continuous sheet or web, such as paper, textiles, plastics, metal, etc. A Waldron web processing range may involve as few as three or as many as twenty unit operations on the continuously moving web. Among the industries to benefit will be metal strip coating in which a comparatively recent development is the continuous coating of steel and aluminium strip, imparting a decorative and durable finish unaffected by normal cold working.

Home and export sales of Simon-Waldron equipment will be handled from Simon headquarters at Cheadle Heath, Stockport, Cheshire. A new department has been formed under the name Coating Plant Department: Mr. C. G. Ahlquist is the director in charge with Mr. E. Rawlinson as sales manager.

## WAVERLEY GOLD MEDAL COMPETITION 1961

RESEARCH is this year sponsoring The Waverley Gold Medal Essay Competition for the ninth year in succession. The Competition is designed to encourage the scientist in the laboratory and the engineer in the production plant to express his views and translate his work into an essay that will be readily understood by other scientists, directors of industrial firms and others interested in science and technology.

The Medal, together with £100 will be awarded for the best essay of about 3,000 words describing a new project or practical development in pure or applied science.

Entry forms can be obtained from the Editor of *Research*, 88 Kingsway, W.C.2.

## NEW COMPANIES

**Jackman Anodised Products**, 36 Southampton Street, W.C.2. February 15. £100. To carry on bus. of metal and alloy makers, anodisers, oxidisers, etc. Harry Jackman, Mrs. Ursula R. Jackman.

**Gullick Brothers (Walsall)**, Wismore Works, Walsall. March 2. £6,000. To carry on bus. of polishers, platers, stove enamellers etc. Ernest C. Gullick, Richard H. Gullick, Annie E. Gullick, Jean P. Gullick, George A. Gullick, Nora W. Gullick.

**Frost and Sons (Metal Spraying)**, Falcon Galvanizing Works, Moxley, Wednesbury. March 3. £100,000. Sidney W. Frost, Harry Frost, Sidney Frost.

## POLYESTER FINISHES

MUCH favourable comment was drawn at the opening of London's tallest hotel, The Carlton Tower, by the furniture in the first floor lounge. This includes white lacquer consoles, sofas with black lacquer frames as well as white lacquer tables, and a white lacquer and brass console over 8 ft. long. Designed by John Siddeley from American originals, they had to be adapted to the needs of the hotel, and the designer took great care to obtain a lacquer of unexampled hardness to give the maximum resistance to wear and tear. He chose Herbolux polyester finishes made by Jensen and Nicholson Ltd.

These lacquers give an extremely hard glass-like surface, they are resistant to scoring, abrasion, marking by packing materials, and their impact resistance is especially good.

They will resist continuous pressure at 100/120°C

Herbolux finishes are a combination of unsaturated polyester and styrene. They can be applied, sanded and burnished by mechanical methods and can be supplied for spray application for vertical surfaces, thus avoiding the necessity of designing furniture exclusively for assembly after finishing, although furniture so designed does lend itself more readily to mechanical finishing with polyesters.

## Change of Address

THE Federation of British Rubber and Allied Manufacturers has moved its offices to 19/20 Berners Street, London, W.1. The telephone numbers, Museum 2671 and 0268 remain unchanged.

**Sprayotron Equipment**, 58 Desborough Park Road, High Wycombe, Bucks. March 6. £1,000. To carry on bus. of manufacturers of and dealers in spraying equipment, in particular electrostatic paint spraying equipment, etc. Leonard T. F. Bryan.

**Ciba United Kingdom**, 96 Piccadilly, W.1. March 7. £3,000,000. To acquire not less than 90% of the issued shares in the capital of Ciba Laboratories, Ltd., Ciba (A.R.L.) Ltd., and Ciba Clayton Ltd. and the whole or any part of the issued shares in the capital of Clayton Aniline Co. Ltd., and to act as the parent company of such companies, etc. Directors not named.

From the Register compiled by Jordan & Sons Ltd Chancery Lane, London, W.C.2.

## SURFACE CORROSION AND CORROSION CONTROL

"WE are somewhat horrified by the tendency in parts of the paint industry to use built-in obsolescence to sell their products . . . the approach whereby corrosion engineers are made to believe that their schemes are obsolete because some new resins or pigments have become available. We know of no single surface coating which will show outstanding performance in all cases," said R. A. Hartley, B.Sc., A.R.I.C., M.C.I.C., technical manager of the Canadian associate company of International Paints Limited, in a paper "Surface Coatings for Corrosion Control" read before the Hydraulic Power Section of the Canadian Electrical Association (Eastern Zone).

"A very much more satisfactory

approach to the problem," he continued, "is to use the best coating scheme which experience and research has shown to be the most satisfactory for these conditions."

In his paper Mr. Hartley gave a practical assessment of the properties and performance of both conventional and recently developed coatings such as vinyls, epoxides, and polyurethanes.

A separate section, entitled "Friction Losses," was devoted to the advantages and problems of internally coating conduits, pipelines and penstocks. Reference was made to the results of the research of various workers on the subject, and examples were given of the saving in cost by coating with materials that have a smooth finish.

### 33rd ANNUAL MEETING OF LEAD INDUSTRIES ASSOCIATION

"LEAD'S New Frontiers" will be the theme of the 33rd Annual Meeting of the Lead Industries Association to be held on May 2 and 3, 1961, at the Drake Hotel, Chicago. One session will be devoted to the latest developments that are keeping lead abreast of the rapid pace of modern technology. These will include the lead zirconate-titanate "spark pump" ignition system for internal combustion engines, pearlescent pigments for use with plastics and on metals, lead-containing sound barriers and ship hull damping compounds, ultrasonic cleaning, thermo-electric devices, nuclear shielding for ships etc.

At another session Research Director S. F. Radtke will report on the substantial progress being made by the Association's own expanded research programme in its search for new markets and products. The secretary and staff will also describe the stepped-up educational, advertising and technical service programme of the lead industry. Economic and technical reviews of the more important established lead products like storage batteries, delivered by industry leaders, will form a part of this session.

The opening session will be held jointly with the American Zinc Institute and will include papers of broad interest to both industries on national and international developments and the general economic outlook.

### Exhibition of Ultrasonic Equipment

A SPECIAL Exhibition of industrial ultrasonic equipment and capacitance stud welding, was recently held by Kerry's (Ultrasonics) Ltd., in conjunction with the Midlands Electricity Board.

The equipment on view included: *Ultrasonic cleaning equipment* where the cleaning fluid is vibrated at high frequencies giving an exceptional cleaning technique avoiding the disadvantages of high temperature, high corrosive cleaning and expensive hand labour.

*Ultrasonic machining equipment.* This equipment has been designed for drilling any configuration in glass, ceramics, tungsten carbide, silicon, germanium and precious stones, with a drill bit of soft steel.

*Percussive high capacitance stud welding equipment.* This equipment may be used for stud welding on material as thin as 0.020 in., and on the reverse sides of plated, painted, enamelled and ceramic coated surfaces, without damage.

### U.S. Bentonite on Sale in U.K.

THE Fullers' Earth Union Ltd., of Redhill, Surrey (a member of the Laporte Group) have concluded an agreement with the Archer-Daniels-Midland Company of New York, to act as sole distributor for sales of "Federal Green Bond" bentonite in the United Kingdom. This addition to their present range of British-produced "Fulbond" bentonites will enable the Union to supplement their service to British industries.

### F. J. Stokes Corporation Establishes British Subsidiary

F. J. STOKES Corporation of Philadelphia, Pennsylvania, has established a subsidiary company in Great Britain. Incorporation of F. J. Stokes Ltd., with headquarters in London, has been announced by Francis Dougherty, Jr., president of the 65-year old American firm.

The new British subsidiary will manufacture some of Stokes' wide range of machines and equipment for the plastics, chemical, pharmaceutical, automotive, electronics and food industries. These machines will be available throughout the British Isles, the Commonwealth, and countries of the European Free Trade Association.

Directors of the new organization are Allan A. Hutchings, vice-president in charge of sales, and F. Joseph Stokes, Jr., vice-president in charge of manufacturing for the American company, and Malcolm Scott of London.

James T. Davies, an Englishman and a former sales engineering executive of the American company, has returned to London to undertake the general management of operations for F. J. Stokes, Ltd.

### FORTHCOMING MEETINGS

**Institute of Metal Finishing (London Branch).** "Plating of Zinc-based die-castings" by J. Edwards, at Northampton College of Technology, St. John Street, London, E.C.1. 6.15 p.m.

April 18  
**Institute of Metal Finishing (Midland Branch).** "Now much Polishing is needed?" Discussion, opened by M. A. Price, at James Watt Memorial Institute, Gt. Charles Street, Birmingham, 3. 6.30 p.m. (N.B. Meeting brought forward from May 2—no meeting in May).

**Institute of Metal Finishing (South-West Branch).** Annual general meeting and film show, at the Royal Hotel, Bristol. 7.30 p.m.

April 28  
**Institute of Metal Finishing (Sheffield and North-East Branch).** Meeting at Grand Hotel, Sheffield. 7 p.m. Subject to be announced.

May 2-6  
**Institute of Metal Finishing.** Annual Conference, Llandudno.

May 10  
**Institute of Vitreous Enamellers (Southern Section).** Works visit to Aerograph Co. Ltd.



## TECHNICAL BOOKSHELF



**Practical Anodizing of Aluminium**, by W. W. G. Hübner and A. Schiltknecht. (Translated by W. Lewis). Publ. Macdonald and Evans Limited. (pp. 330, Figs. 187). 55s.

The rapid post-war expansion of aluminium usage has seen an even more rapid development in the quantity of aluminium decorated and protected by anodizing and rapid advances in the early associated technology. There are many who during this period have sought a reliable guide to the theory and practice of anodizing, but have found the only information available dispersed amongst numerous published papers and text books.

This volume was published in German in 1956 and is written essentially for the practical anodizer. Its treatment of the theory of the process is quite superficial, and the whole emphasis, as implied by the title, is on the practical aspects.

The first part of the book deals in turn with chemical oxidation processes, anodizing, chemical brightening, electrobrightening, dyeing, sealing and allied processes. This section is somewhat disappointing in that the details given are limited, and the practical man is left with no guidance as to why particular conditions are chosen. For example, most newcomers to the field would like some explanation as to why different concentrations of sulphuric acid are used commercially, and of the reason for different recommendations as to operating temperatures. The authors confine themselves to recommending a single concentration, namely 231 gm./l. sulphuric acid, although they give some indication of temperature requirements, but no indication of the influence of current density on the rate of film growth under these various conditions.

There is an appreciable section devoted to the layout of plant and buildings and the designing and construction of anodizing equipment. This provides an interesting insight into German practice in this field at the time the book was written, but there are a number of points on which the text is already dated, particularly the introduction of the newer types of rectifiers and of automatic current control apparatus. It will be noticed that some of the materials for construction differ from those employed here, while the use of a water pump to provide a reciprocating movement on an anode rail is

a means not so far encountered by the reviewer in this country. The section on mechanical polishing is one of the most satisfactory portions of the book. As far as the reviewer is concerned, it appears to be one of the few attempts that have been made to examine polishing processes and techniques on a scientific basis, and to discuss the various types of equipment and material from basic principles.

The beginner in anodizing will also welcome the section on jiggging which illustrates quite a number of methods of jiggging that can be used on anodized work. This is a field where new ideas are being continually developed and where the experience and know-how of the anodizer is far more important than technological excellence. Some anodizers will be intrigued and surprised to study the set of illustrations (Figs. 176, 177) in which great care has been taken to provide internal air agitation to hollow articles, such as coffee pots, etc.; a practice rarely encountered in this country.

This book will be of interest and assistance to those without prior anodizing experience, but to the experienced anodizer will be of little help, although he may profitably spend time comparing and contrasting information given by the authors with that of his own experience. Those about to enter the anodizing field for the first time will find a number of points of interest in the text, but will have cause later to amend it in the light of their own experience and techniques. The book would have been more welcomed if it had appeared in English at about the same time as it appeared in German.

A. W. BRACE.

**Metal Polishing.** Buckart, Silman and Draper. Robert Draper Ltd., Teddington, Middx. 267 pp. 205 illus. 60s. Postage 1s. 9d.

This book consists of nine chapters covering 255 pages. It has 205 illustrations covering both polishing plant and apparatus, and various surface photographs and micrographs.

The matter covers the polishing of both metals and non-metals, for example, plastics, wood, glass, ivory, etc.

The book is undoubtedly useful and is probably the best attempt that

has been made so far to write a text-book of polishing.

It deals with practically all the processes that are used at the moment, including automatic polishing machines and barrel polishing and many of these are described in detail. There is also some theoretical matter incorporated, although this could well be expanded.

The book suffers, perhaps, in being a little dis-jointed, some processes being described in two or three places. Some of the descriptions are also not entirely consistent; for example, in one place tripoli is described as being composed of diatoms (p. 30), while in another (p. 129) it is described as being hydrated silica. In fact, the tripoli that is used today does not, as supplied, show any diatoms but is generally a calcined natural product containing silica, etc.

There are also one or two misprints, a very obvious one being the Table on page 60 where the surface speed of polishing wheels is shown as being 600-900 ft. per min. instead of 6,000-9,000 ft. per min. It is understood that the publishers are issuing a correction slip for this. Also on page 202 it is stated that the optimum peripheral speed is 100-300 ft. per sec., which can be obtained at 2,000-25,000 r.p.m., with a 12 in. diameter wheel, instead of 2,000-2,500 r.p.m.

The data on polishing plastics, wood, glass, etc., is of considerable interest as these substances have not yet been dealt with to any large extent in similar works.

The book as a whole is interesting and is well worth reading by anybody who is concerned with polishing in any form. It is hoped, perhaps, that in further editions some of the points raised may be tidied up and a more concise story presented.

E. A. OLLARD.

### "LIFT-O-MATIC" AGENCY

Electro Plating Equipment Co. Ltd., of 120/130, Parchmore Road, Thornton Heath, Surrey (telephone Livingstone 7793) are now handling the Lift-o-matic Plant, manufactured by Plating Chemie N.V., of Holland, and all enquiries for this equipment should be addressed to the above company.

This company was founded in 1954 and became part of Sonic Engineering and Equipment Ltd. in 1958 until recently, but is now trading again as a separate company.

## Trade and Technical Publications

The Geigy Co. Ltd., have issued a booklet giving brief details of their chemicals for metal treatment, but say that individual circulars giving more comprehensive details of the various products are available on request from the Development Division, Rhodes, Middleton, Manchester.

A recent issue of The Cellon Bulletin, published by Cellon Ltd., Kingston-upon-Thames, Surrey, contains, among other interesting items, an article on Electrostatic Spray Finishing.

Aerostyle Ltd., of Sunbeam Road, North Acton, London, N.W.10, have re-printed their Water Wash Spray Booth brochure as a more comprehensive pamphlet showing the increased range of spray booths.

A further improvement is that these standardized spray booths can now be made in a wide range of sizes, the sections being made in either 2 ft. or 2 ft. 6 in. panels, thus enabling a wide range of booths every 6 in. in width.

A new catalogue issued by KABI (Electrical and Plastics) Ltd., Kabi Works, Cranborne Road, Potters Bar, Middlesex, shows the present range of KABI bench assembly trays and storage bins.

The publication "pH Meters by E.I.L." is a fully coloured catalogue describing the complete range of pH meters produced by Electronic Instruments Ltd., Richmond, Surrey.

A number of accessories are shown, such as the E.I.L. pH multi-electrode switching unit, the pH electro-pneumatic converter, pH two-way switching unit and pH alarm unit.

One of the most important features about E.I.L. industrial pH meters is that they are designed to work with all conventional recorders or recorder-controllers marketed by the leading manufacturers at home and abroad. Page 11 lists well-known firms, with their trade-marks: Bailey Meters, Elliott, Record, Sunvic, Fielden, Evershed, Honeywell, Negretti & Zambra, Ether Ltd., O.T.I.C., Bristol's, Taylor, Foxboro, Electroflo.

The booklet ends with "What is pH?" which gives a simple explanation, which can be understood by the semi-skilled control operator. This is followed by a view of the E.I.L. research laboratories and notes

on design, and photograph of the fleet of servicing vans and a typical installation.

The Copper Development Association, 55, South Audley Street, London, W.1., issue every month a publication entitled "Copper Abstracts." These abstracts are based on a survey of more than 120 technical and other periodicals and of such relevant books as may appear from time to time. The April issue, No. 26, contains over 50 abstracts, a proportion of which are included in the heading "Finishing and Plating."

A technical data sheet dealing with "Promex" cold applied strippable coating has been issued by Croda Ltd., Goole, Yorks.

Promex is a complementary product to the "Croccl" range of thick film hot-dip strippable plastic coatings for anti-corrosive protection. Some customers found that it was

not always possible to dip items particularly large ones, into "thick film" coatings applied from heated baths; neither are thick films always desired. Such items can now simply be dipped in cold liquid Promex and allowed to dry.

Promex forms a tough, elastic skin on metal surfaces which protects them against corrosion, abrasion, scuffing, etc., under a wide range of conditions, e.g. during storage or delivery to customers. It is sufficiently adherent to the metal surface to "stay put," but can easily be removed by peeling. Standard Promex is clear, but it can be pigmented with aluminium powder, or dyed green, red or blue. It is applicable by brushing, spraying or dipping.

Hanson-Van Winkle-Munning Co., Matawan, New Jersey announce the availability of a new edition of its Electroplating Processes Bulletin. The 24-page, two colour new edition, EP-103, is an expansion of EP-102 issued last year. Nineteen plating and other metal-finishing processes and procedures are described. Solution preparation, type of deposits, operating conditions, equipment required, and applications are listed for each of the processes.

### I.M.F. MIDLAND BRANCH DINNER

THE Midland Branch of the Institute of Metal Finishing held a dinner on February 24 at the Grand Hotel, Birmingham. The illustration shows Mr. C. R. Darby (Chairman of the Midland Branch) and Mrs. Darby with Mr. C. Wharrad (Chairman of the Metal Finishing Association) and Mrs. Wharrad.

A number of prizes were presented for events during the evening, all expenses associated with these being paid by local firms interested in the plating industry.



# Latest Developments

## in

# PLANT, PROCESSES AND EQUIPMENT

### Recirculating Ovens

**G.** AND R. Gilbert (Industrial) Ltd., Hackbridge Road, Hackbridge, Wallington, Surrey have recently increased their range of standard ovens for stoving, drying, curing, pre-heating, baking etc., by including a new series of recirculating ovens with three forms of heater and thirty-six standard sizes.

The primary advantages of recirculating ovens over other types of batch oven are floor level loading, without recourse to pits or ramps, more uniform temperature conditions, the ability to handle more densely packed loads without creating cold spots, and in many cases to cut process times by reason of faster heat transfer and quicker removal of solvents and vapours from the working space.

Fig. 1 shows a typical direct gas-fired unit from this range, showing the heater and fan assembly mounted on top of the oven. This arrangement, is usually preferred, as it occupies the minimum floor space. Where headroom is limited, however, the heater may be placed at floor level on one side of the oven.

Directly fired ovens have a Neat gas burner firing into a stainless-steel combustion cylinder mounted in an insulated steel heater body, recirculating air being drawn through the body of the heater mixing with the products of combustion before passing to the oven over a special type of hot gas fan. Full thermostatic and safety controls are supplied with these ovens as standard equipment.

Electrically heated ovens have a series of metal sheathed magnesium oxide or Steatite-insulated heater elements fitted into an insulated steel casing. Deflectors are used to provide increased surface area for heat transfer. Elements are normally connected back to burners fitted in a closed casing formed on top of the heater. The standard series ovens do not have flameproof equipment but this can be supplied in some cases. Ovens are provided with thermostatic control and H.R.C. fused isolators.

The sectional construction used enables installation in buildings with restricted access and allows widths of 5 ft., 6 ft., and 7 ft., heights of 6 ft. 6 in. and 7 ft. 6 in. to be built up in 4 ft., 5 ft., 6 ft., 8 ft., 10 ft. and 12 ft. depths. Larger ovens and steam or oil-fired units employing basically standard sections can be built up to order.

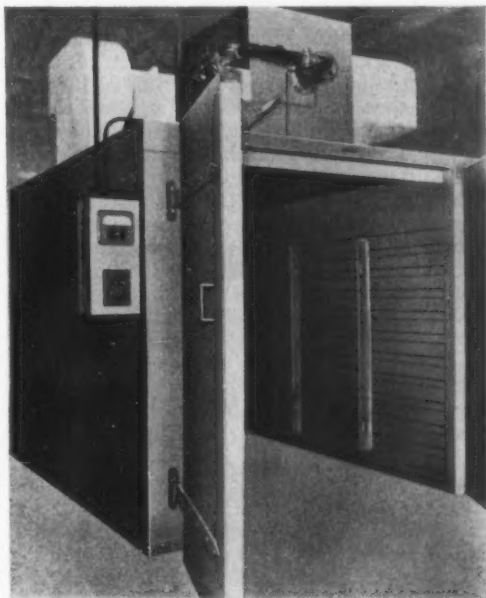


Fig. 1—Recirculating Oven

### New Attachment Measures Surface Finish of 1-mm. Ball

**T**HE prototype of a new development that enables the surface roughness of a 1-mm. diameter ball to be measured and recorded accurately, in 0.02 of a micron, was shown by the Taylor, Taylor and Hobson Division of Rank Precision Industries Ltd., at the Leipzig Fair. It is the small ball surface measuring attachment for use with the Talysurf Model 3 surface measuring instrument, which has been designed with a view to production testing, in answer to the widespread demand for the measurement of high-speed precision bearings.

The attachment consists of a stationary pick-up unit in which a stylus is mounted underneath a conical seating. The ball to be tested is placed in the seating where it is then rotated by a friction drive, controlled from the standard Talysurf gearbox. The ball is given the same surface velocity as is normally applied to the traverse of the Talysurf pick-up in its conventional form. This permits

both graphs and average readings to be obtained from a standard Talysurf 3.

The range of balls catered for is 1 mm. to 25 mm. Graph readings are recorded with magnifications up to  $\times 50,000$  (2.5 mm. on paper representing 0.00005 mm).

Another new development is the new model 105 Talysurf, which will measure the surface finish inside cylinders of 6.0 mm. diameter to a depth of 250 mm. It is a portable and relatively inexpensive instrument, suitable for a wide range of workshop and inspection applications. It provides centre-line average readings with 0.75 mm.

Two other new attachments for the Talysurf Model 3 have also been developed. These are the cylindrical datum attachment for dealing with radii of from 50 mm. to infinity by the supply of suitable glass cylinders. This accessory is complementary to the existing radii attachments and deals with surface finish on both convex and concave radii and (2) an adaptor for fitting a side acting Talymin 3 gauge head to the Talysurf Model 3 gear box. The Talymin 3 gauge head gives a magnification range of  $\times 100$  —  $\times 5,000$ .

#### Cold Soak Cleaner

THE Pyrene Co. Ltd., Metal Finishing Division, Great West Road, Brentford, Middlesex, have produced "Pyroclean No. 104,"—an emulsifiable paraffin-based solvent-type cleaner of the soak type which is used cold for degreasing work coming forward from phosphating; it is specially formulated for cleaning iron and other metals and may be used in still tanks and in automatic immersion tanks, and is supplied in two forms—a concentrate No. 104C which is normally diluted with paraffin in the ratio of one part to four, and a ready-for-use material (No. 104D), which requires no further dilution or addition. Parts to be cleaned are immersed in the cold solution for 2-15 min. and then thoroughly rinsed through two water lines. The parts should be free from water since this will emulsify the chemical.

#### Audible Thickness Gauge

A PORTABLE, battery-operated thickness gauge manufactured by Metallisation Ltd. of Barclay's Bank Chambers, Dudley, Worcs, is designed specifically for checking the thickness of sprayed zinc and aluminium coatings. The principle of operation is that after setting the instrument to indicate the minimum thickness required, an exploring head is quickly moved over the coated surface and an audible warning is given of areas which are below the required thickness. Thicknesses may be determined between 0.002 in. and 0.012 in.

#### Copper Plating Process

RECENT work in the Albright & Wilson (Mfg.) Ltd. Laboratories at Oldbury, near Birmingham, has resulted in the development of a new electrolyte based on pyrophosphate for copper plating. The "Pyrobrite" bath is said to give far better results than any other copper plating method.

The company manufacture and supply all the constituents necessary for the "Pyrobrite" bath, the main feature of which is the new PY61 addition agent. This ensures fully bright deposits over a wide current-density range. The bath is claimed to have good levelling characteristics and to produce close-grained, smooth deposits, which do not finger-mark or spot out. The bath plates faster than acid or cyanide solutions under normal operating conditions, because higher current densities are possible, and has excellent throwing power. Because there is no cyanide content there is no need for fume extraction and no problems associated with effluent disposal. The solution itself is only mildly alkaline and does not attack light alloys to any appreciable extent. The process is suitable for electroforming, and is claimed to provide a dense, smooth deposit without the formation of nodules. Because the deposit is non-porous, a relatively thin layer forms an efficient stop-off medium during nitriding or carburizing of steel.

Another application for the process is in copper plating prior to bright nickel and chromium plating of zinc-based die-castings and steel. The levelling properties of the solution often make it possible to eliminate mechanical polishing of the base metal before nickel plating directly onto the copper. A 'strike' solution based on copper pyrophosphate is available for articles made from steel.

#### Etching Primer to Reduce Fire Risk

FEDERATED Paints Ltd. of Glasgow, have produced an etching primer, Strathclyde Clean Conditions Primer PA-22, which is claimed to substantially reduce fire risk. The new product is a modified version of Primers PA-20 and PA-21 which were designed specifically to come within the conditions laid down by the U.K. Atomic Energy Authority; the solvent used in these primers had a low flash point to ensure rapid drying and consequently fire precautions necessary were difficult to enforce, particularly in confined spaces such as ducting. The new primer incorporates a primer with a flash point of over 73°F., and is not, therefore, highly inflammable. Drying time has been slowed down slightly, although it still dries, under normal conditions, in less than an hour.

### Portable Air Compressor

**B**.E.N. Patents Ltd., High Wycombe, Bucks, have produced another new unit in their range of portable air compressors — the "PD three" unit (Fig. 2). This is a versatile, fully mobile, multi-purpose unit specifically designed for general use in car showrooms, small garages and workshops, also for spray painting, house decorating, creosoting and agricultural purposes — spraying insecticides etc.

The single cylinder air-cooled compressor (which also incorporates a moisture separator and pressure gauge) displaces 2.58 cu. ft. at 100 lb per sq. in. It is direct coupled to a  $\frac{1}{2}$ -h.p. electric motor and the unit is secured to a welded tubular steel frame mounted on generously proportioned rubber tyred wheels. A tubular steel handle welded to the frame is fitted with carrier brackets for the hose and cable (supplied as standard fittings).

As a special feature of the "PD three," the motor incorporates a Thermotrip cut-out device which effectively protects it against overload or burn-out.

A "Vari-Pressure" control valve permits an instant choice of operating pressure for any required duty by the one-hand manipulation of a milled adjusting nut.

A B.E.N. IM spray gun can be supplied as an extra and hangs on the hook at the back of the tubular steel handle, as illustrated.

Fig. 2—Portable Air Compressor



### Metallized Materials

**A** COMPREHENSIVE range of metallized materials is now available from E. S. and A. Robinson Ltd., Redcliffe Street, Bristol. The company have installed a high vacuum metallizing plant in Bristol and intensified their previous research work into high vacuum metallization and into the pre- and post-treatments of a wide range of materials for processing under this technique. The first major development is the announcement that the company are now producing, and using commercially, a range of metallized films and textiles.

The rolls of metallized materials immediately available include non-toxic unplasticized p.v.c. films — for example, the "improved clarity" Craytherm manufactured by Greenwicks Plastics Ltd. — in calipers ranging from 0.001 in. to 0.020 in., the heavier calipers being particularly suitable for vacuum-formed trays and packs; cellulose acetate film up to 0.0075 in. for boxes or for box lids and edges; cellulose acetate (0.001 in.) for laminating to paper or board for labels, swing tickets and general display and packaging work; cellulosic films which can be coated on the unmetallized side with heat seal lacquer for use as wrappers; and various polyester and polyamide films.

### Adhesive for joining polyurethane or polyether foam to fabric, wood, metal

**A** NEW adhesive, Evo-Stik "Impact" Adhesive 633, consisting of a one-part rubber/resin formula, has been developed by the Industrial Adhesives Division of Evode Ltd., Stafford, to provide a strong resilient bond when joining polyurethane or polyether foam to materials such as fabric, wood, metal etc., without affecting the microcellular structure of the foam.

The adhesive provides a strong, efficient bond for microcellular foam to itself without causing any hardening effect at the joint, is water and oil-proof, and resistant to high temperatures.

All surfaces to be joined must be thoroughly clean, e.g. metal degreased or wiped with trichlorethylene and dust and other foreign matter removed from foam.

Application is by brush to both surfaces to be joined, taking care that an even film of adhesive is spread overall. Allow to dry for approximately 10-30 minutes and then press the two materials together using firm hand pressure over the whole area. At this stage an immediate strong bond will be obtained so that the joined components can proceed unhindered in the course of production.

If the adhesive is to be applied by spray gun or roller coater, it is recommended that three parts of the adhesive should be mixed with two parts of

*Continued in page 164*

## Plant, Processes and Equipment

(Continued from page 163)

Evo-Stik Cleaner 191 before use to give the desired consistency. Alternatively, a material of the correct viscosity for spray or roller coating known as Evo-Stik "Impact" Adhesive 633/S can be obtained.

Further details may be obtained from Evode Limited, 82 Victoria Street, London, S.W.1.

### Treatment for Factory Floors

**T**HE Magnus Chemical Co. Ltd., Salisbury Road, Industrial Estate, Uxbridge, Middlesex, have recently been appointed sole concessionaires for the U.K. and part of Europe to cover the marketing and distribution of a mineral-oil and grease absorber called "Drener-Dri." The compound is said to absorb oil and grease up to one third of its own weight, and it is only necessary in use to sprinkle the compound over an oily floor and allow it time to soak up the oil. It can be used again and again until saturated. The compound is dust-free and will not support combustion even when saturated with oils and other flammable liquids.

The company will supply samples if required or arrange to demonstrate the material.

## Classified Advertisements

Prepaid rates: FIFTEEN WORDS for 7s 6d. (minimum charge) and 4d. per word thereafter, or 24s. per inch. Box number 2s. 6d. including postage of replies.

### SITUATIONS VACANT

#### ENGINEER for CHEMICAL SALES

A young mechanical engineer is required as a sales representative by Albright & Wilson (Mfg) Ltd. Duties are to develop the sales of plant and equipment connected with Albright & Wilson's metal finishing processes. The post is located in London and extensive travel in the U.K. will be required to visit customers and to co-operate with the existing sales force. H.N.C. in mechanical engineering is a minimum qualification, and experience in a firm concerned with metal finishing will be an advantage. Age 25-30 or older if suitably experienced. The Company operates non-contributory pension and housing schemes.

Apply to Staff Officer,  
Albright & Wilson Ltd.,  
Group Personnel Dept.

\* Knightsbridge Green, London, S.W.1. Reference No. 70.

### SITUATIONS VACANT—Cont.

#### A MECHANICAL ENGINEER

is required in the Metal Finishing Section of the Central Engineering Department of ALBRIGHT & WILSON (MFG) LTD., Oldbury, Birmingham. This section deals with all aspects of plant for electro-plating and chemical polishing from design to installation and commissioning at customers' premises, and the engineer will be responsible for all phases of contracts in this field. The minimum qualifications for the post are H.N.C. Mechanical Engineering, together with some basic knowledge in one or more of the following: electrical engineering, chemical engineering, chemistry or metallurgy. Previous experience in the field of electroplating is not essential, but would be a distinct advantage. Preferred age range—26/36. The Company operates non-contributory Pension and Housing schemes. The Metal Finishing side of the business continues to expand rapidly. Applications, quoting reference no. 66, should be addressed to:—

Staff Officer,  
Albright & Wilson, Ltd.,  
Group Personnel Dept.

\* Knightsbridge Green, London, S.W.1.

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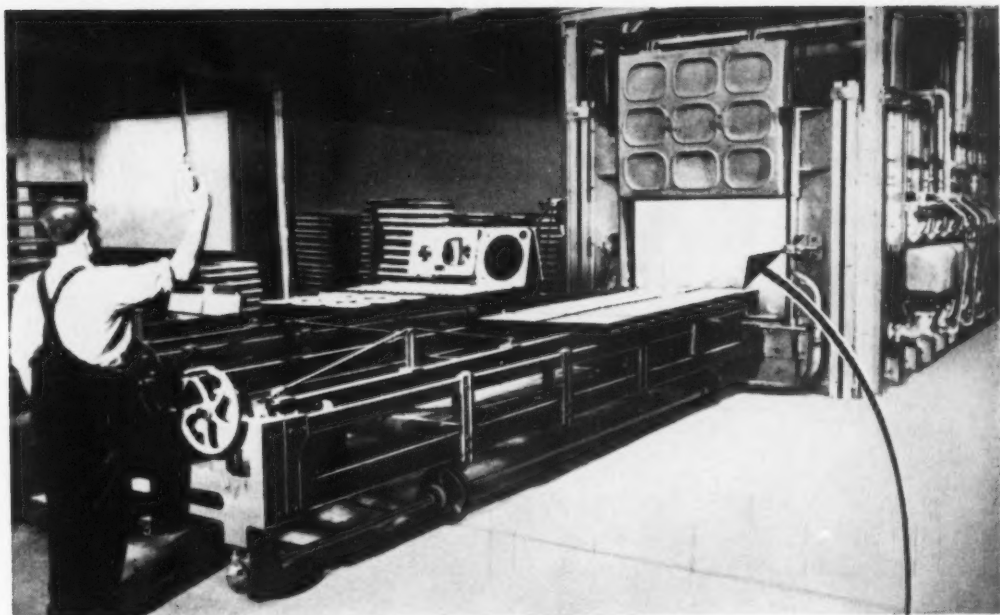
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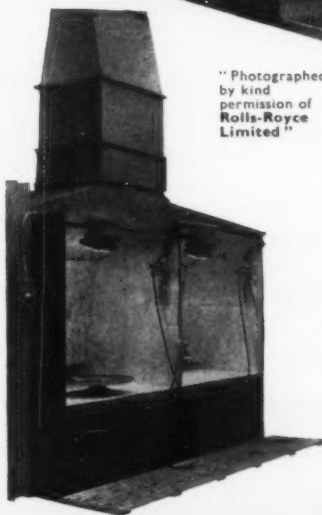


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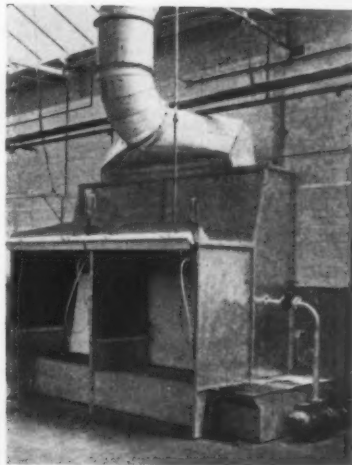
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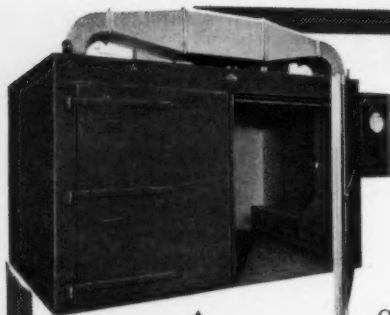
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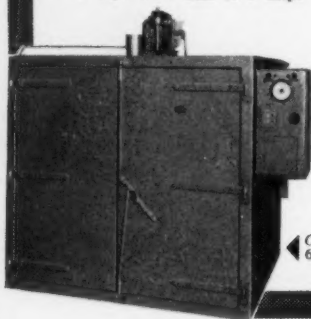
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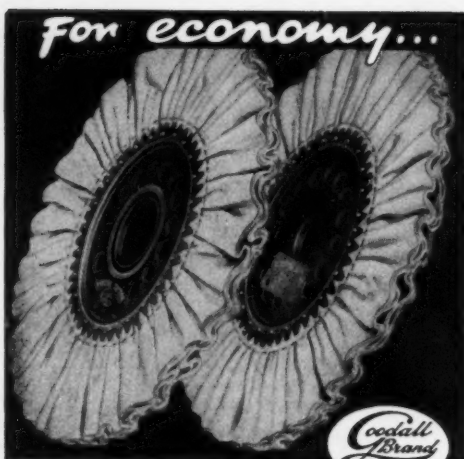
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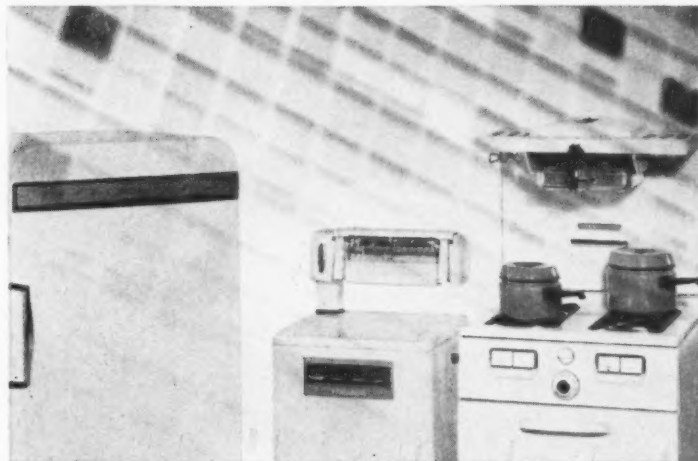
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